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A NONCONTACTING SCANNING PHOTOELECTRON EMISSION TECHNIQUE FOR BONDING SURFACE CLEANLINESS INSPECTION

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16. ABSTRACT

Molecular contamination of bonding surfaces can drastically affect the bond strength that can be achieved and therefore the structural integrity and reliability of the bonded part. The presence of thin contaminant films on bonding surfaces can result from inadequate or incomplete cleaning methods, from oxide growth during the time between cleaning (such as grit blasting) and bonding, or from failure to properly protect cleaned surfaces from oils, greases, fingerprints, release agents, or deposition of facility airborne molecules generated by adjacent manufacturing or processing operations.

Required cleanliness levels for desired bond performance can be determined by testing to correlate bond strength with contaminant type and quantity, thereby establishing the degree of contamination that can be tolerated based on the strength that is needed. Once the maximum acceptable contaminant level is defined, a method is needed to quantitatively measure the contaminant level on the bonding surface prior to bonding to verify that the surface meets the established cleanliness requirement.

This paper describes a unique photoelectron emission technique for the nondestructive inspection of various bonding surfaces, both metallic and nonmetallic, to provide quantitative data on residual contaminant levels. The technique can be used to scan surfaces at speeds of at least 30 ft/min using a servo system to maintain required sensor to surface spacing. The fundamental operation of the photoelectron emission sensor system, which is commercially available, is explained and the automated scanning system and computer data acquisition hardware and software, which was developed by MSFC, are described. Illustrative data are provided relative to sensor and system performance for both bonding test specimens and full scale hardware (SRM) inspection applications. Current capabilities and limitations of the use of this methodology are addressed and plans for future development and application are summarized.

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INTRODUCTION

The performance of many critical components for the Space Shuttle and other flight hardware depends on the quality of bonding achieved during fabrication of the component. An example of a major Shuttle element where quality bonding is crucial to performance, reliability, and safety is the Solid Rocket Motor (SRM). Inadequate bonding of the rubber insulation to the case could result in exposure of the D6AC steel case to the hot gas from the burning propellant and result in burn-thru which could be disastrous. Also, low strength bonds between the various nozzle parts could significantly affect SRM flight success.

Because of the high reliance placed on bonded parts and the wide variability in strength that is often observed, a comprehensive program was initiated by the Materials and Processes Laboratory of the Marshall Space Flight Center (MSFC) to investigate ways of improving bonding process control during the manufacture of critical SRM hardware in order to improve overall bond quality and reduce within-part and part-to-part variability in bonding strength. The process control parameters that affect bonding integrity include adhesive variability, storage, mixing, pot life, contamination of the bonding surface, surface preparation, adhesive application, and curing. Thus, all of these were included in the program. In addition, since bonding process control must be an inherent part of the overall manufacturing process, it has to be specific with respect to the sensitivity of the adhesives, bonding surfaces, and subsequent bonds to the environments (moisture, thermal, contamination) encountered during the manufacturing flow. For this reason, the program was implemented as a joint endeavor between MSFC and the Wasatch Division of Morton Thiokol, manufacturer of the SRM.

Of all of the above parameters that affect bonding, contamination is probably the most insidious and least understood. The presence of thin molecular films on bonding surfaces can drastically affect the strength of some bonding systems. These films can result from inadequate or incomplete cleaning, oxide growth during the time between cleaning (e.g., grit blasting) and bonding, or from failure to properly protect cleaned surfaces from oils, greases, fingerprints, release agents, or deposition of airborne molecular species generated by adjacent manufacturing or processing operations. These films may or may not be uniformly deposited on a large area bonding surface which can lead to variation in bond quality across that surface. The thickness and chemistry of the film, its interaction with adhesive, and the adherends, and the subsequent response to the curing process can all affect the degree and level of bonding achieved. Often these contaminants are invisible, making detection and quantitative measurement difficult and expensive. Thus, in order to eliminate or minimize contamination as a threat to bond integrity, strict contamination control of bonding surfaces is required. To assure the proper degree of control, required cleanliness levels must first be determined. Second, a methodology must be established for the uniform cleaning of the surfaces to the established levels and maintaining them at these levels from the completion of cleaning to the initiation of bonding. Third, prebonding inspection of the surfaces is required to verify that

they have been properly controlled. This bonding process contamination control scenario is illustrated by means of the contamination control triangle shown in Figure 1.

Previously, the rigorous application of this approach to contamination control of bonding surfaces was severely restricted due to the lack of a fast, cost effective method for quantitatively measuring contaminant levels on hardware bonding surfaces after cleaning and prior to bonding to verify compliance with established cleanliness requirements. Since such a method was needed for the SRM bonding improvement program, a development effort was initiated by MSFC which resulted in the technique described in this paper.

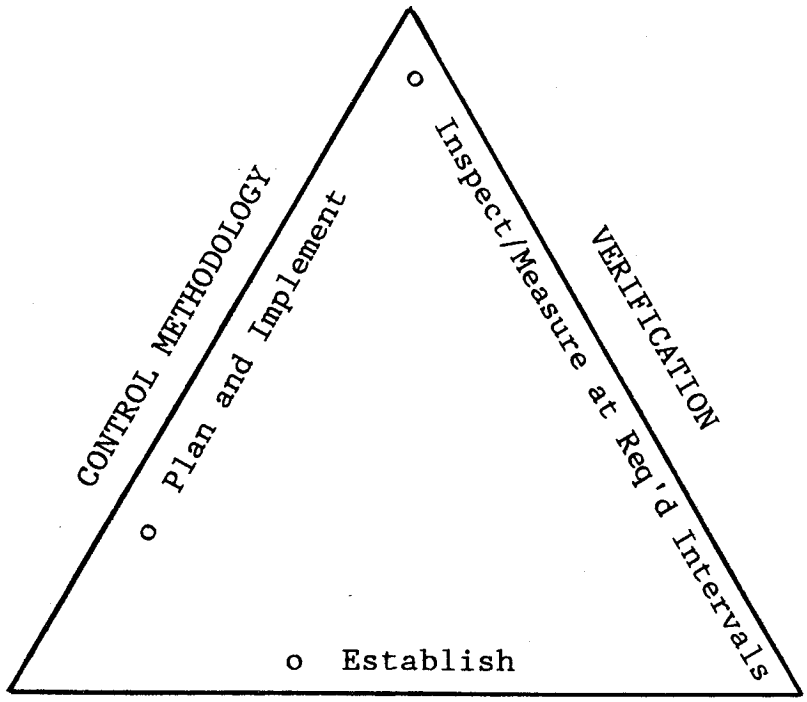

Figure 1. Contamination control triangle.

TECHNIQUE DESCRIPTION

The prototype system that was developed for scanning SRM case segments to detect and quantify residual containant levels is shown schematically in Figure 2. The system (acronym ConScan for contamination scanning) consists of a commercially available photoelectron emission [or alternately, optically stimulated electron emission (OSEE)] sensor, whose description and theory of operation will be given later; a servo controlled sensor position controller for maintaining the proper sensor to scanned surface distance; encoders for providing sensor position coordinates (height, angle) relative to the case; a revolution counter; a computer data acquisition, analysis, display and storage system; and software specifically developed for this application. For scanning of cylindrical articles such as the SRM case, the article is placed on a turntable and the position controller, with sensor(s) mounted, is installed on a

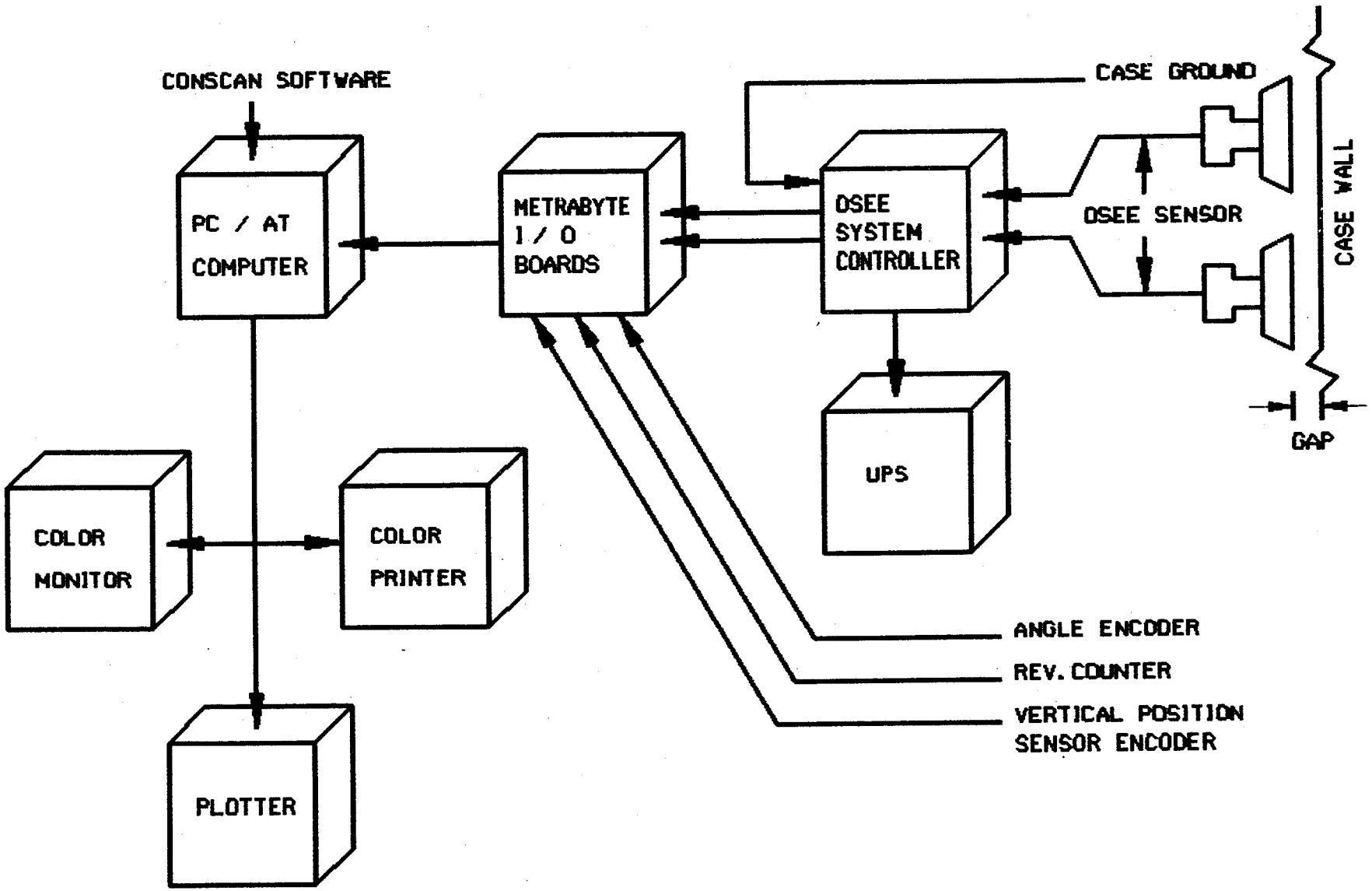

Figure 2. Prototype case surface contamination measuring system schematic.

translation stage such as a robot arm which moves the sensor vertically as the article to be inspected is rotated by the turntable. The article can be eccentric, since the position controller continuously senses by means of a proximity detector the distance between the contamination sensor and the surface being scanned and maintains, through a servo loop, the required spacing. The setup for the scanning of the SRM case is shown schematically in Figure 3. In this application, the sensor/position controller [Fig. 3(a)] is mounted on a hydraulically controlled cage that can be moved vertically as the case is rotated.

Two scanning modes can be accommodated, barber pole or manual indexing. In the barber pole mode, the sensor is moved vertically at a constant rate during part rotation, thereby providing a spiral inspection. Spiral spacing can be controlled from zero to the percentage overlap desired by adjusting the rate of vertical translation relative to the rotational speed. The second mode of operation, manual indexing, is useful for rescanning of previously scanned areas. For instance, if a complete scan is made using the barber pole mode and a contaminated area is detected, the vertical and angular coordinates of that area can be obtained from the data display and the sensor can be translated to that vertical position and the part rotated to the required angular position. In this manner, the contaminated area can easily be located for cleaning. After cleaning, just the area cleaned can be reinspected by indexing at desired increments through the area to verify that the area had been cleaned to the required level.

As the part is scanned, the sensor output is continuously input to the data system where it is correlated with the sensor height and the angular position of the part, thereby providing a readout of the sensor as a function of position relative to the part. Using previously developed calibration data relating sensor output to contaminant level, the sensor readings can be translated into contaminant levels. Accept/reject values based on contamination versus bond strength tests can be displayed on the color monitor by assigning colors to the ranges of sensor output corresponding to these values. Thus, as the part is scanned, the reject areas will be displayed in the specified color in real time on the monitor. The ConScan software developed for analyzing and displaying these data is user friendly in that a question/answer format is employed which simplifies selection of scan mode, scan parameters, accept/reject levels, etc. Once a scan is completed, the operator can at once determine whether the part is acceptable or whether further cleaning is required. If cleaning is required and the part is rescanned, the original data are retained in the computer memory and an update is generated for the area rescanned. Thus, archival records are obtained to document the inspection. If a hard copy of the scan data is needed for review, one can be made with the color printer [Fig. 3(b)]. A plotter is used to graph the actual sensor output for verification of proper functional operation. The successful application of the above system depends on (1) a knowledge of the fundamental performance capabilities and limitations of the photoelectron emission contamination sensor, (2) calibration of the sensor output as a function of contaminant level on the specific surface to be inspected, and (3) the effect of contaminant level on bond quality. These factors along with relative illustrative data are discussed in the following sections.

SENSOR CHARACTERIZATION

The contamination sensor used is commercially available from Photo Acoustic Technology, Inc. (PAT), Newbury Park, California, which advertises it as a surface quality monitor. Since it is a patented device (T. Smith, Inventor), and there is no

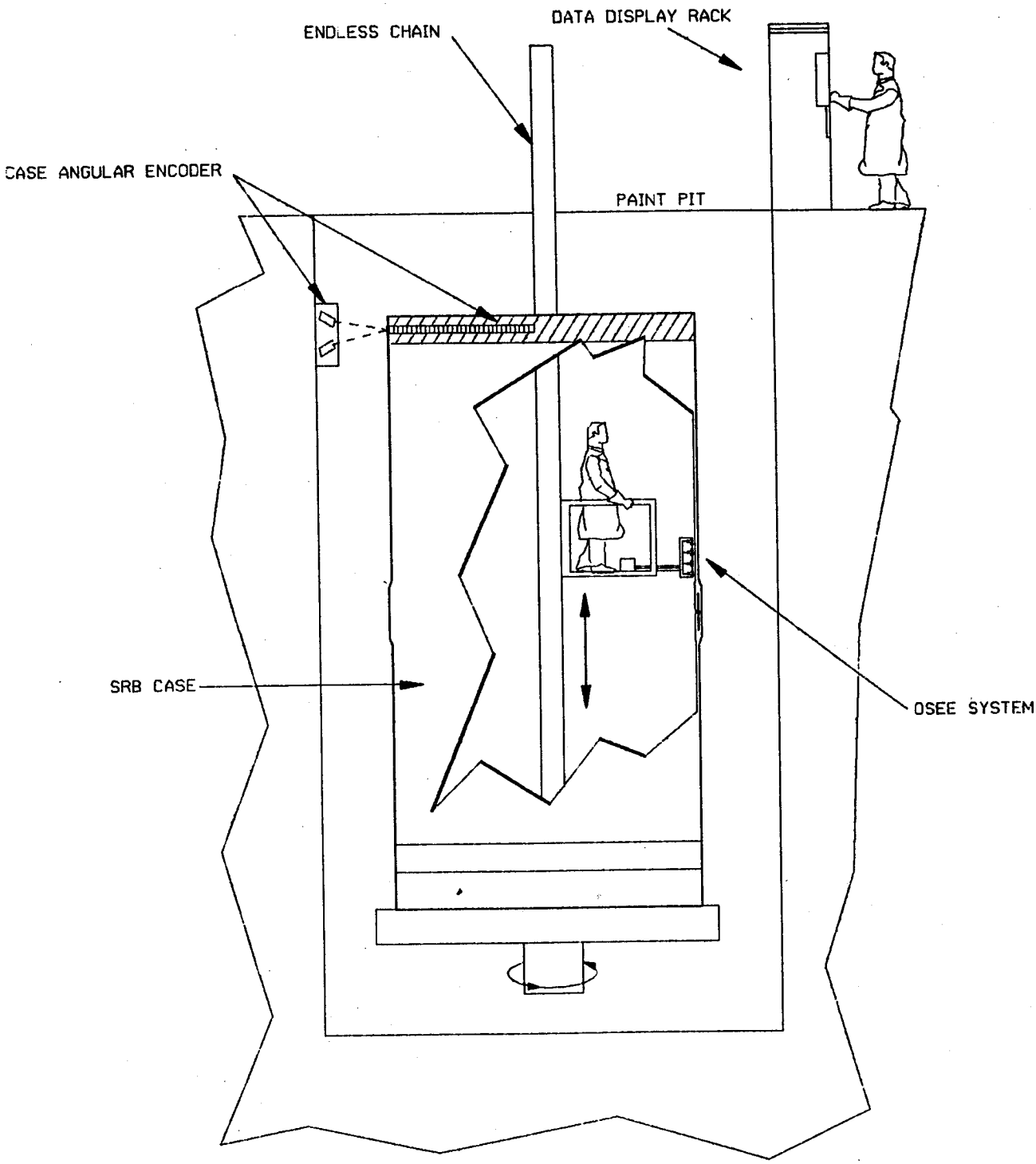

Figure 3. Surface contamination scanning concept using OSEE system.

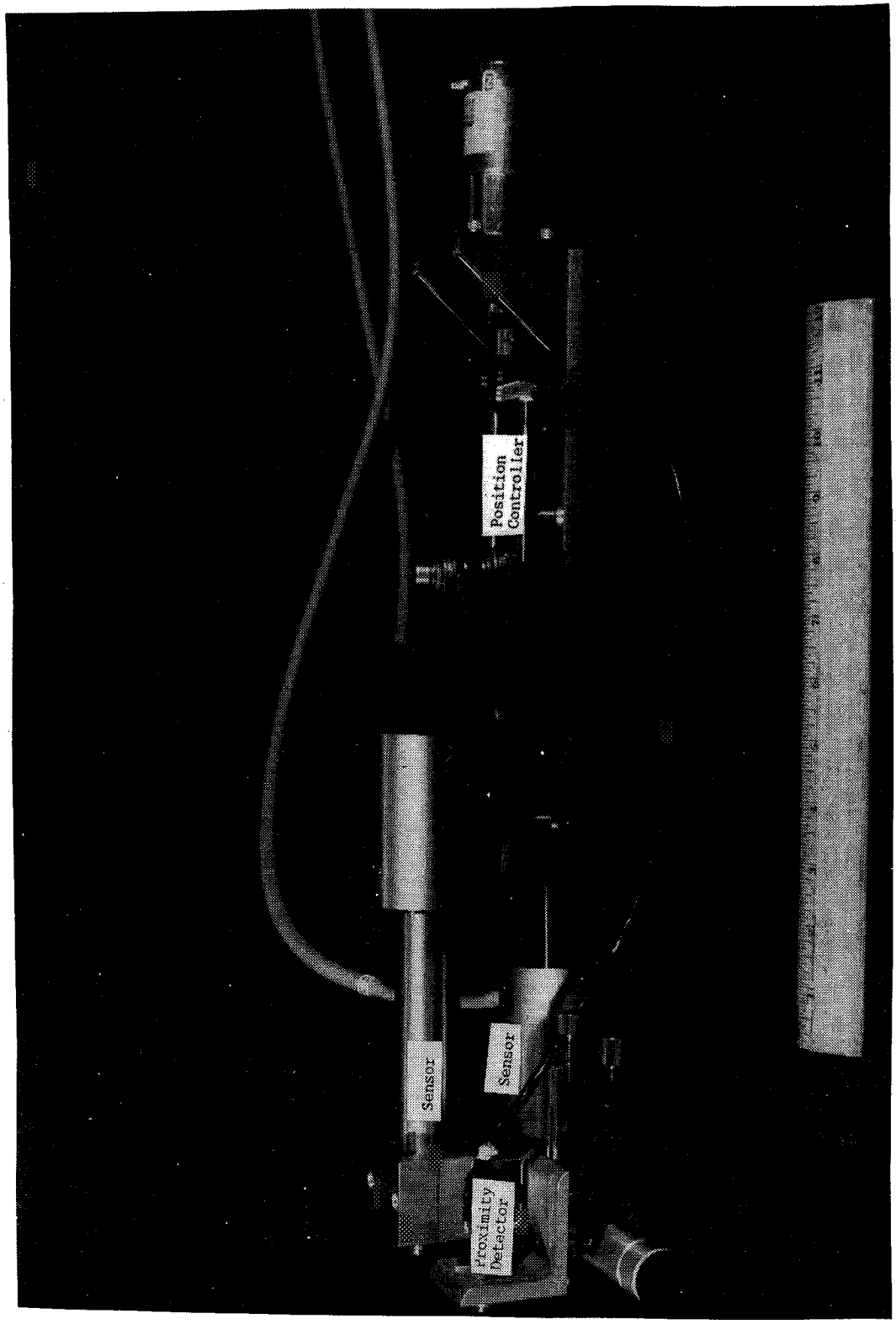

Figure 3(a). OSEE sensor/position controller for SRM case inspection.

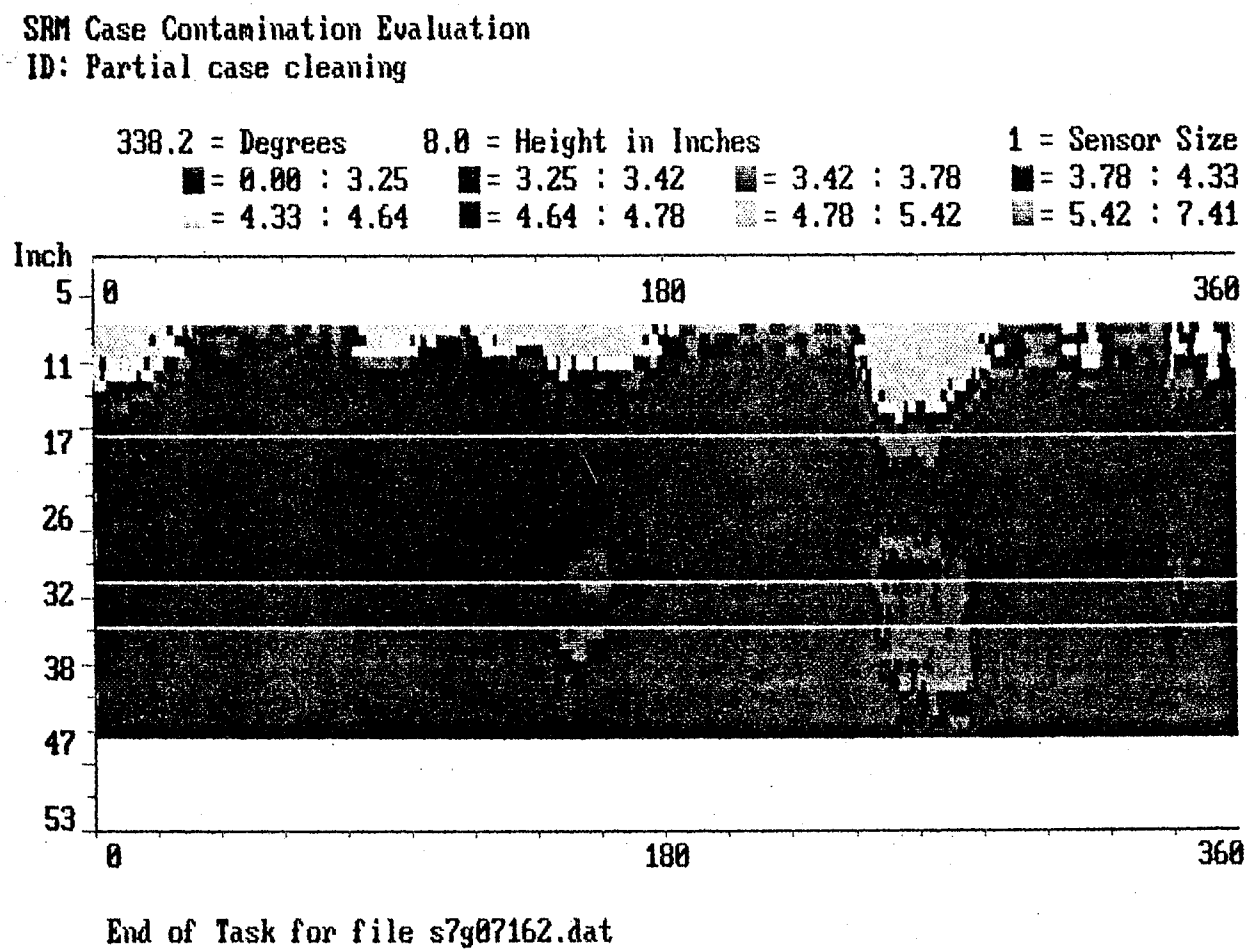

Figure 3(b). Example of hard copy of ConScan data.

other licensee, it is the only instrument of its kind on the market. This type of sensor was selected based on the results of previous development work [1,2] sponsored by NASA and other investigations [3,4,5] which showed that the photoelectron emission technique, the principle of operation for the PAT sensor, has the capability of providing quantitative thin layer contamination data for various types of contaminants and surfaces, e.g., silicone on aluminum, oxides on metals, and oil on silicon wafers. Of course, as for any analytical tool, it has certain operational sensitivities and limitations which must be understood for proper utilization of its inherent capabilities for both laboratory and production applications and environments.

Theory of Operation

It is well known that when metals or certain other surfaces are illuminated with ultraviolet (UV) light with the proper wavelength (energy) electrons are emitted from the surface. The process by which UV photons interact with the surface to produce electrons is known as photoelectron emission (PEE) or optically stimulated electron emission (OSEE). Smith [4] showed that the emitted and subsequently scattered electrons can be collected across an air gap by a biased collector and measured as a current [4]. If the intensity and energy of the UV light and the surface-to-collector distance are held relatively constant, changes in the measured photocurrent (which is on the order of 10^{-10} to 10^{-12} A) can provide information about the surface, e.g., electronic structure, composition, and chemistry. Any contaminant on the surface, depending on its own photoemission characteristics, can either enhance or attenuate the inherent emission from the clean surface. In simple electronic terms, the clean surface is a current generator, and a non-photoemitting contaminant acts as a resistance because the current is attenuated by interactions between the electrons and the contaminant. The thicker the contaminant, the higher the resistance and consequently the greater the decrease in the measured current.

Figure 4 (a through d) depicts schematically the photoemission process and the effect of various thicknesses of contaminant (t) on the photocurrent (I). If the contaminant is photoemitting, e.g., Conoco HD-2 grease, then the contaminant can act as a resistance and an electron generator. Whether the collected current is attenuated or enhanced will be controlled by the contaminant film thickness and the relative emission characteristics of the contaminant with respect to the substrate surface. If the thickness is great enough to totally absorb the UV so that no UV photons reach the substrate, then the measured current is due only to those electrons emanating from the photoemitting contaminant. If some UV photons reach the substrate, and the film thickness is such that the resulting substrate electrons are not totally absorbed, then the measured current (I) will be the sum of the current from the substrate (I_s) and that due to the contaminant (I_c). The case of a photoemitting contaminant is illustrated in Figure 4(e).

The PAT photoelectron emission sensor (Fig. 5) consists of a detector head that contains a UV source (Hg-Xe lamp), a collector electrode with biasing circuitry, and amplification electronics mounted in a small housing which is connected to a controller (Fig. 6). The controller provides the power for the lamp and the electronics for processing and displaying the signal from the detector. The signal flow block diagram is shown in Figure 7. Since the currents measured by the instrument are very low (picoamperes), a significant amount of specialized circuitry is involved in keeping the signal level above the noise and in providing stable measurements. The sensor is connected to the controller by means of a shielded coaxial cable which is available in

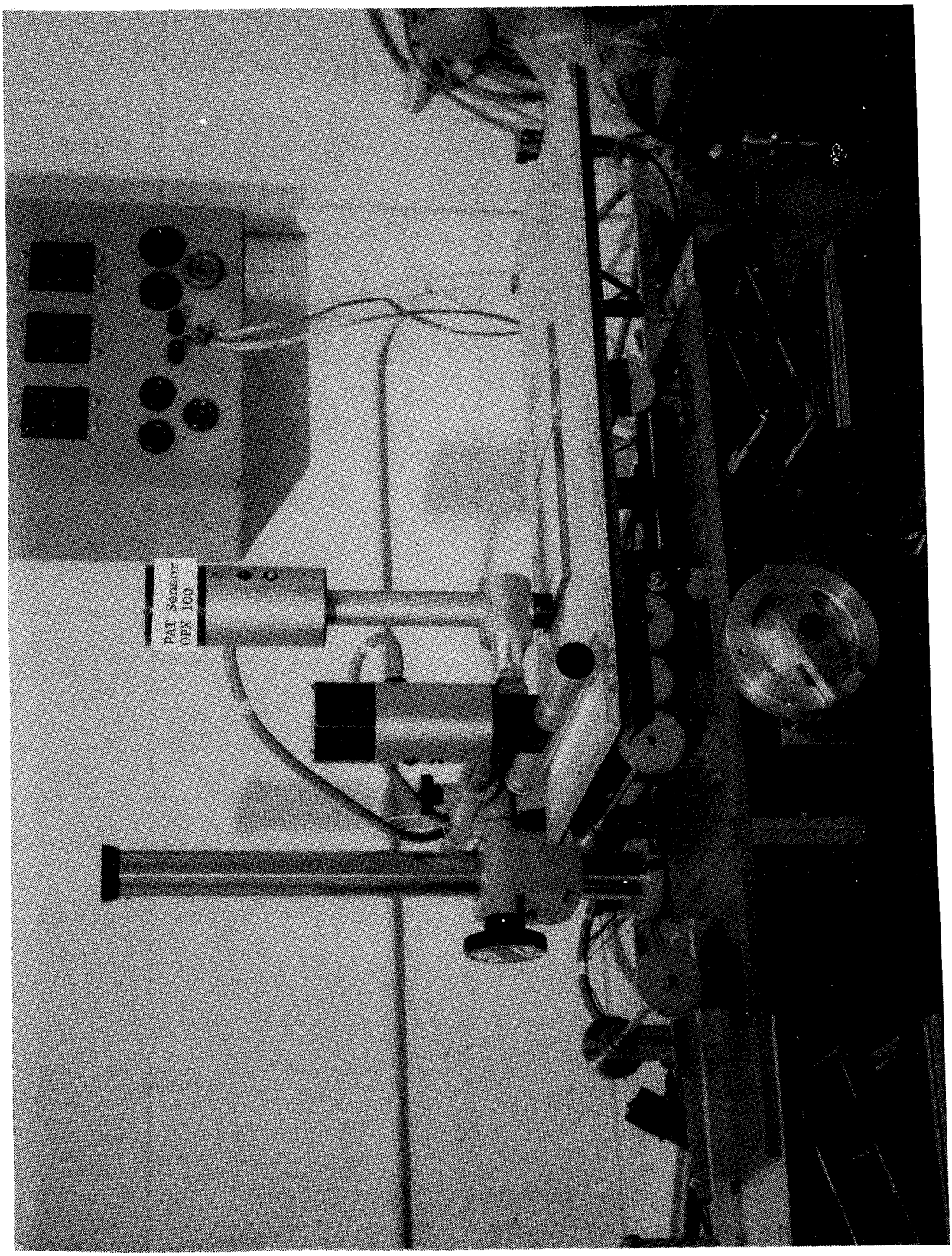

Figure 5. PAT sensor.

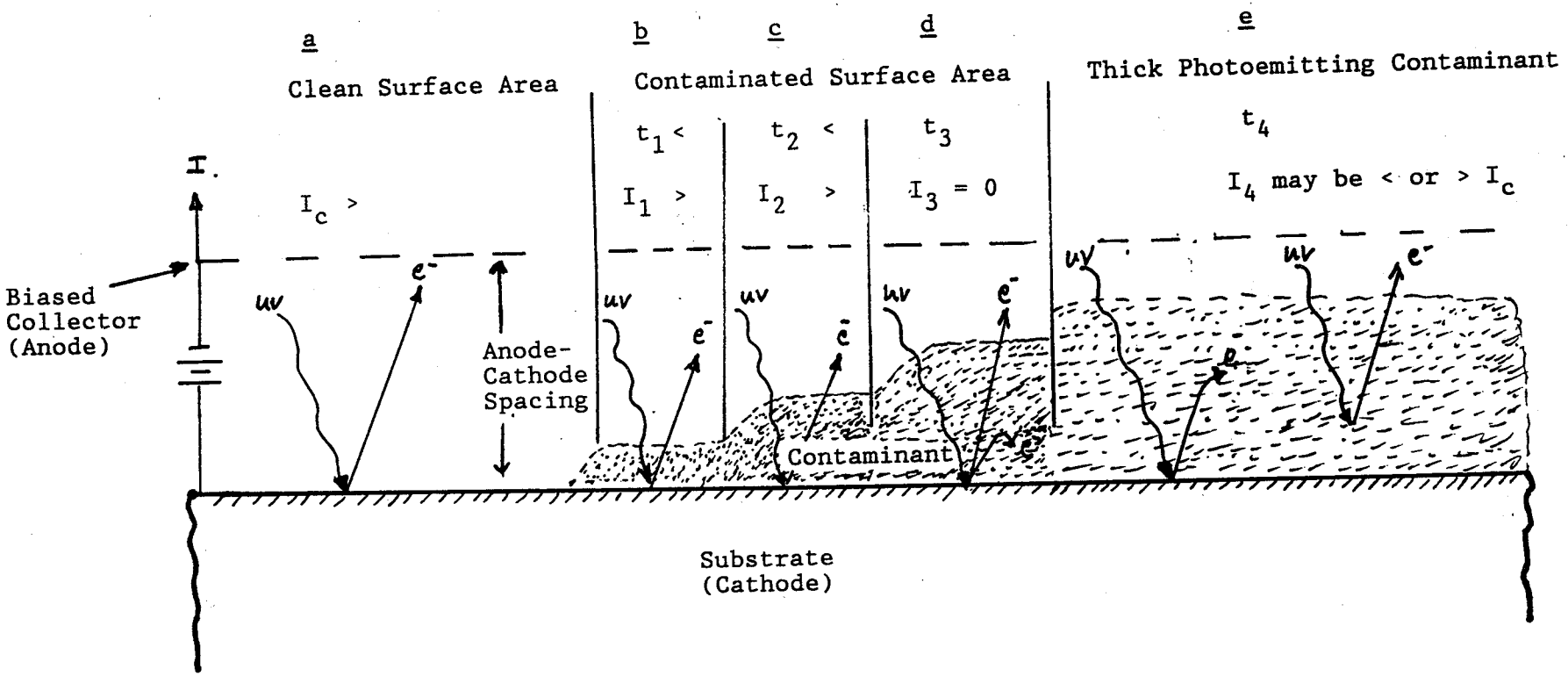

Figure 4. Effect of contaminant thickness on photocurrent.

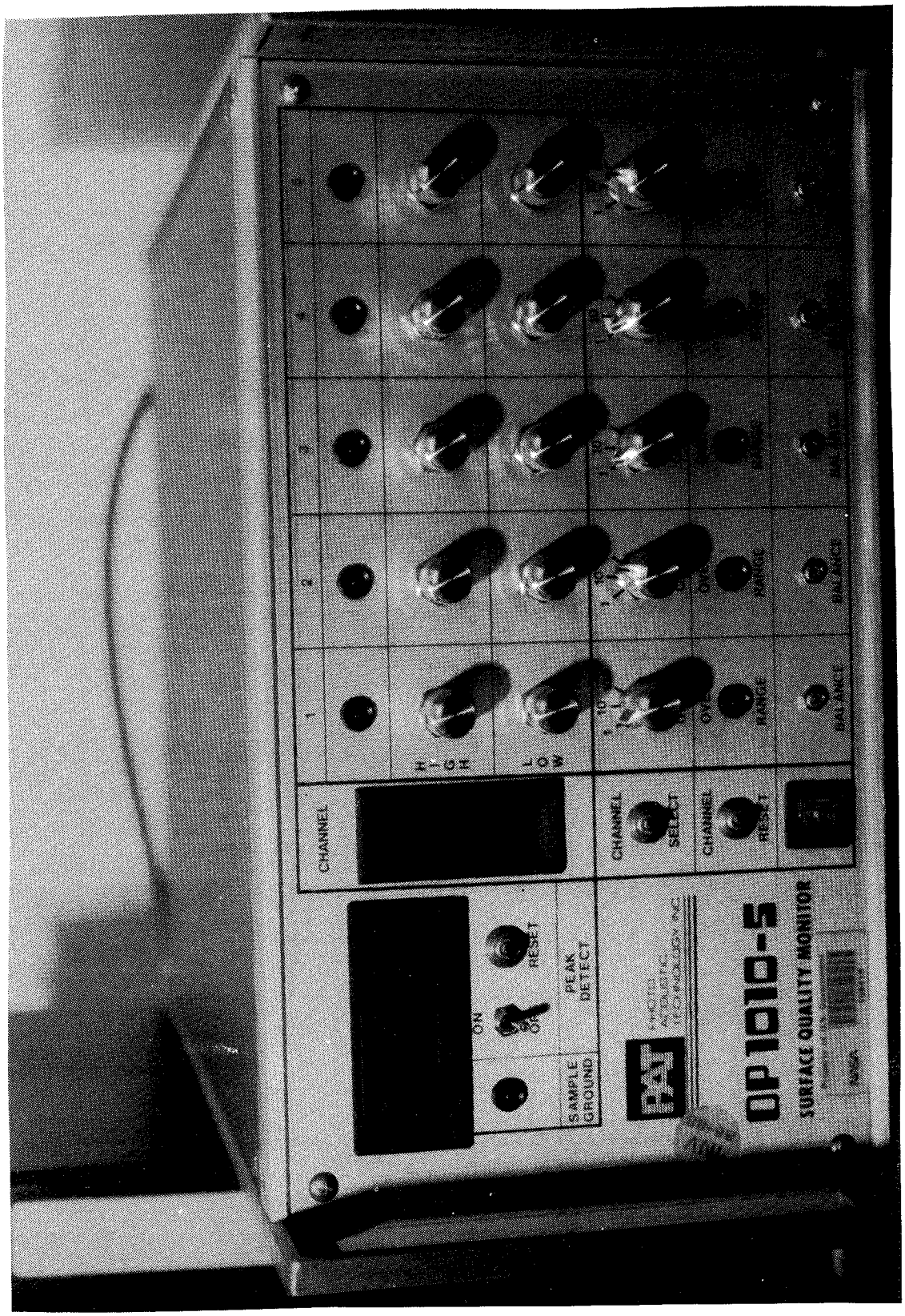

Figure 6. PAT sensor controller.

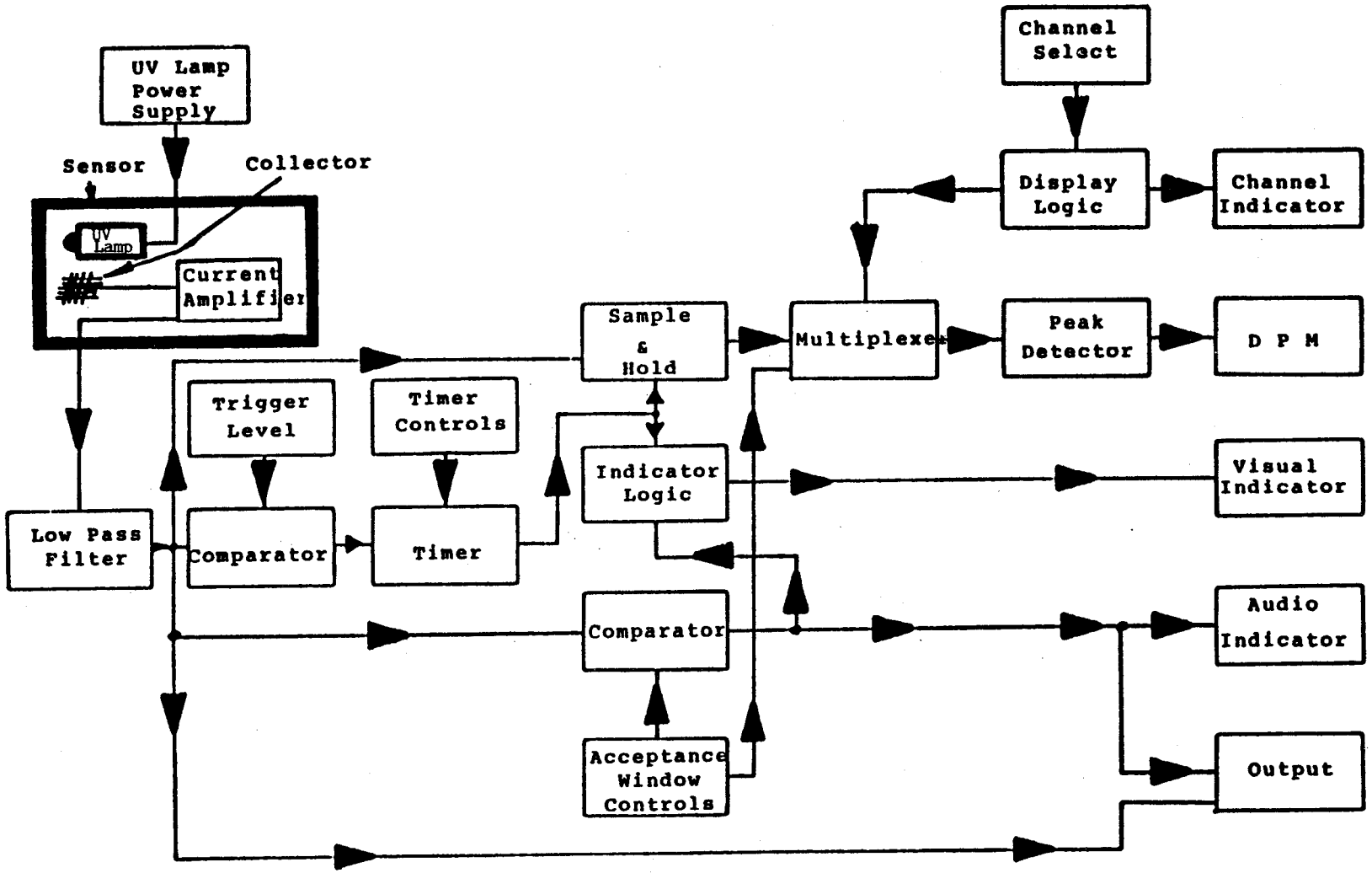

Figure 7. Signal flow block diagram.

lengths up to 100 ft. A sample ground cable connection is provided which must be connected to the part/material being inspected. This is in turn connected to an earth ground. Sample-to-ground resistance should be less than 10 ohms. In theory, the operation of the sensor is straightforward. It is calibrated by measuring the photocurrent from a clean surface and then from identical surfaces with controlled amounts of a contaminant to obtain a correlation between the sensor output (digital display or analog signal) and the contaminant level. By making measurements in the same way on another surface of unknown cleanliness and invoking these data, the residual contaminant level can be determined for that surface. In actual application of the sensor for making quantitative, repeatable measurements, there are several complicating factors which can affect the measurement, the most significant of which are discussed in the following sections.

Effect of Sensor to Surface Spacing

The sensor output is dependent on the spacing between the sensor and the surface being measured. This sensitivity is basically due to three factors. First, as the sensor moves further away from the surface, the UV intensity on the surface decreases and fewer photoelectrons are produced since the magnitude of the photocurrent is directly proportional to the number of UV photons impinging on the surface per unit of time. Second, with increasing distance the photoelectrons have to travel further, resulting in electron loss due to an increasing number of collisions with the ambient gas molecules. Third, the electric field strength that is developed by the bias voltage between the collector (anode) and the surface (cathode) is dependent on the distance between the two ($E = V/D$). Thus, as the distance increases the collection efficiency decreases due to the decrease in the field driving the electrons toward the collector. The integrated effect of these three factors on the output from a PAT Model OPX100 Sensor measuring a vapor deposited chrome surface is shown in Figure 8. As shown in the figure, the sensitivity starts dropping significantly at a distance (gap) of approximately 0.275 in. Thus, if the sensor is used in a noncontact scanning mode, the maximum spacing should be specified at no more than 0.25 in. so that a margin in position control is available for maintaining high sensitivity.

Surface Variation Considerations

Materials used in engineering applications rarely, if ever, have perfectly smooth and homogeneous surfaces. In fact, considerable effort is made to provide specific finishes for particular applications. Also, alloys are generally used instead of pure metals to obtain improved properties. To complicate things further, the alloys are subjected to different heat treatments to enhance particular characteristics. These processes can lead to different sensor responses.

Table 1 shows the results of OSEE sensor measurements made on a GAR S22 microfinish comparator. The results are interesting. For example, the response from a 500 microinch (μin.) milled finish was 20 percent lower than from a 63 μin. milled finish but a 125 μin. profiled finish has the same response as the 63 milled finish. Not only is the average surface roughness a factor but also the mechanical process by which it is attained. In general, however, as the roughness increases using the same process, the response decreases, probably due to the increased scatter of the UV light and the larger emission angles for the electrons which reduces collection efficiency.

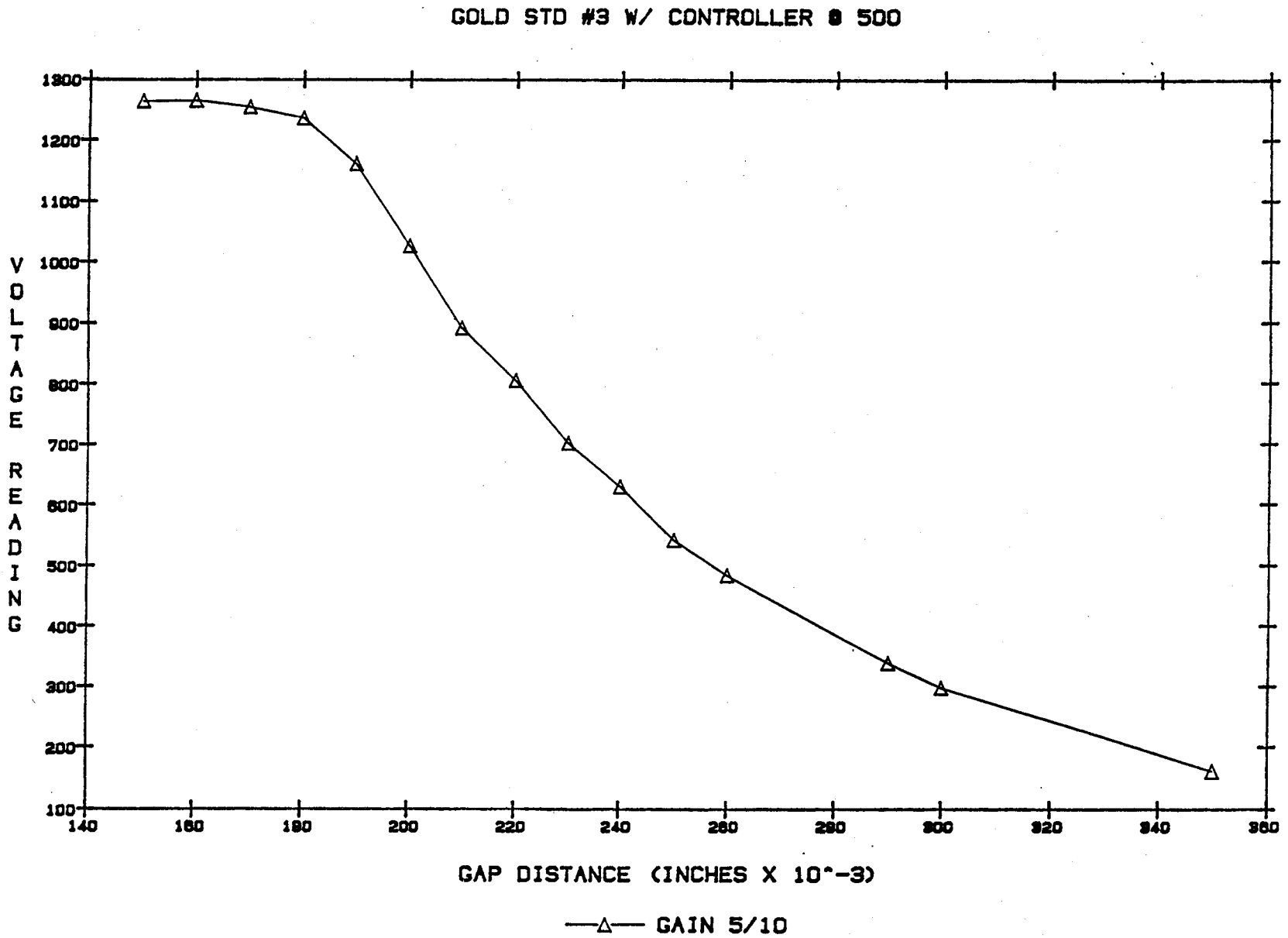

Figure 8. OSEE response versus sensor to surface (gap) distance.

TABLE 1. EFFECT OF SURFACE FINISH ON OSEE SENSOR RESPONSE

Surface Roughness Height (μ-in)	Relative Sensor Output*
8 Ground with Periphery of Wheel	658
16 Ground with Periphery of Wheel	700
32 Ground with Periphery of Wheel	688
2 Lapped	870
4 Lapped	835
8 Lapped	750
63 Milled	800
125 Milled	824
250 Milled	640
63 Profiled	863
125 Profiled	799
250 Profiled	520

*Measured on a GAR S22 Microfinish Comparator

Oxide formation on alloys is usually not uniform over the surface which causes a variation in sensor output. Generally, oxides are not photoemitting at the maximum UV energy of 6.7 electron volts (eV) available from the sensor UV source (nickel oxide is an exception). Thus, freshly grit-blasted aluminum will give a very high response whereas aluminum with a thick oxide layer will give a very low response since the oxide attenuates the photocurrent. It is therefore important to calibrate the sensor response for each specific surface (material, finish, heat treatment, processing) to be measured. Also, it should be expected that a variation in sensor response will be observed when aluminum or other oxide forming materials are abraded since the oxides are difficult to totally remove.

Response to Various Materials

The sensor response to various materials generally depends on the magnitude of the photoelectron work function of the material relative to the maximum usable UV energy reaching the surface from the sensor lamp. The principal high energy peaks from the lamp are at 1849 Å ($\sim$6.7 eV) and 2535 Å ($\sim$5 eV). For relatively large gaps, the 1849 Å line will be severely attenuated due to interaction with ambient oxygen molecules. Hence, it should be assumed that $\sim$5 eV is the maximum energy available for measurements. Thus, if a material has a work function less than 5 eV, it should produce a measurable photocurrent. Table 2 lists published photoelectron work functions for several materials. Note that all of these are less than 5 eV except for aluminum oxide. Both conducting and nonconducting materials can be photoemitting, examples of which are the materials in Table 2. Other examples include the epoxy primer (EA 9228), carbon phenolic, and glass phenolic materials used in the fabrication of the SRM nozzle. In addition to these, successful measurements have been made on graphite/epoxy and fiberglass/epoxy. Some materials which do not exhibit a measurable response include Teflon, glass, and magnesium fluoride. The fact that Teflon is not photoemitting is fortunate in that it can be a detrimental surface contaminent for bonding and is therefore detectable with the OSEE sensor since it will attenuate the photocurrent from the bonding surface.

In general, it appears that measurements can be made on most of the materials of engineering importance, which means that the OSEE measurement technique has great utility.

Scan Rate

The sensor response time is dependent on the gain setting used, which is adjustable from 1 to 10. Very good response time is achieved for gain settings of 6 or less. As the gain is increased beyond 6, a notable drop in response time occurs. With the proper gain setting, high scan rates are possible. For example, at a gain setting of 5 on a PAT OPX200 (6 in. x 0.25 in. inspection area), highly repeatable measurements were made at scan rates of 1 in./sec to 7.5 in./sec on a large test article composed of several surfaces with highly different photoemission characteristics (Fig. 9).

Low scan rates usually are not desirable for some surfaces such as epoxy primers and paint since the sensor output will decay with time of exposure. This effect is discussed in some detail in references 1 and 2. It is recommended that surfaces exhibiting this decay be scanned at a minimum rate of 1 in./sec unless only the peak reading is being recorded.

TABLE 2. ELECTRON WORK FUNCTIONS (EV) OF VARIOUS MATERIALS

Material	Photoelectric Work Function (EV)*	
Zn	3.08	
Mg	3.68	
Fe	3.91	
Be	3.92	
Ti	3.95	
Pb	3.97	
Ta	4.05	
Cd	4.07	
Al	4.08	
Pt	4.09	
Mo	4.15	
W	4.35	
Si	4.37	
Cr	4.37	
Ag	4.73	
C	4.81	
Au	4.82	
Ni	5.01	
Al_2O_3	$\sim$ 8	Ref. 4
NiO	$\sim$ 5	

*Ref. Handbook of Material Science, Volume 1, 1974, CRC Press.

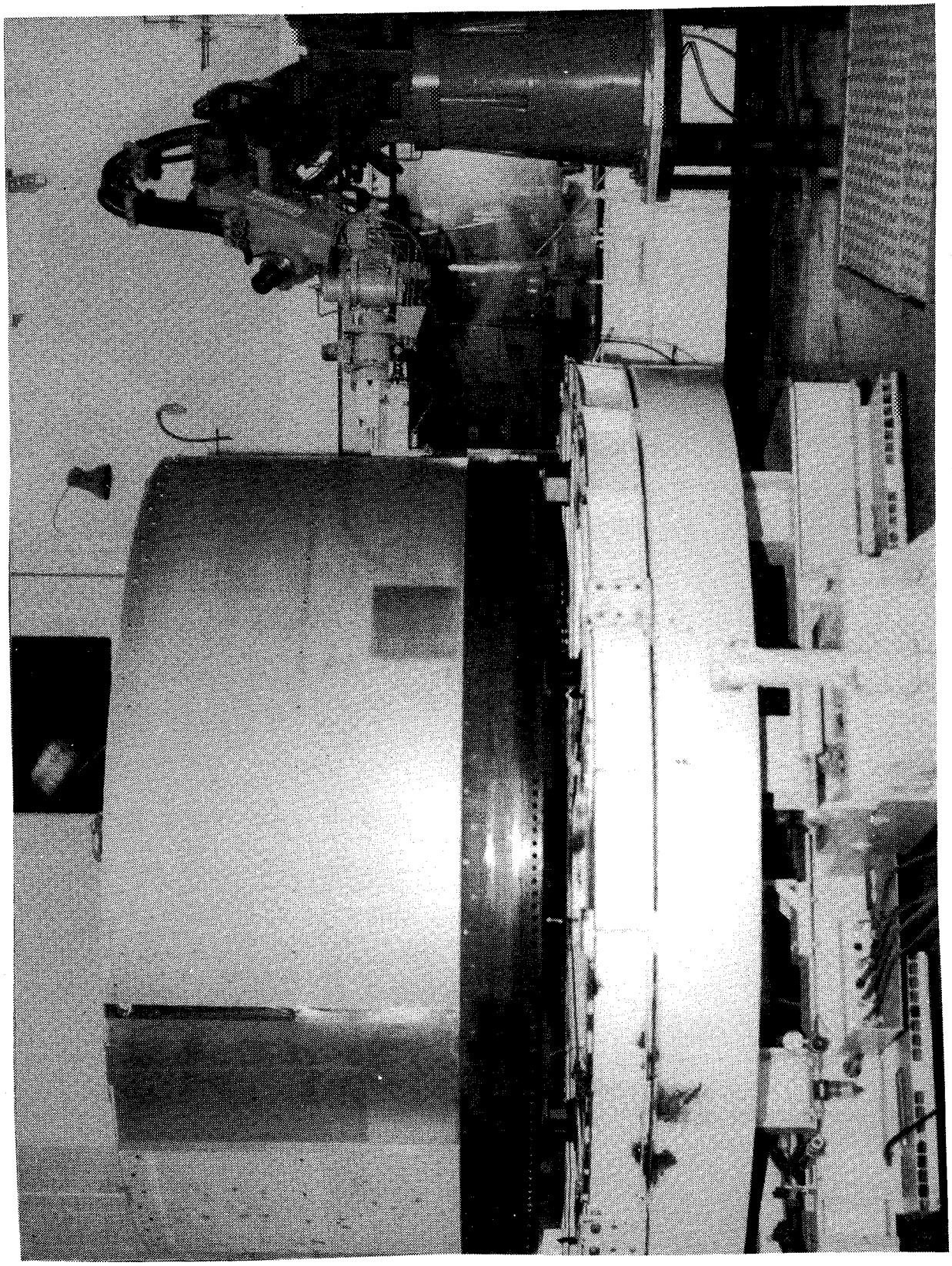

Figure 9. OSEE sensor scan rate test article.

Repeatability and Stability

During the MSFC investigations reported in this paper, excellent repeatability and stability generally was demonstrated by the OSEE sensor. However, some problems were encountered that require discussion. It was noted that the sensor would be highly stable for days or even weeks and then a shift in output magnitude would occur. The instrument was being used in a laboratory whose power was relatively stable but fluctuations were known to occur when large welding machines were used in another part of the building in which the laboratory was located. Based on this observation, it was decided to power the instrument through a line regulator. This essentially eliminated the problem. However, when the line voltage dropped below the line regulator limits, the problem repeated itself. A line monitor was used to record out-of-limit changes in voltage and frequency. So long as the voltage and frequency are within limits, no problem is encountered with the instrument. In a production environment, major power fluctuations are frequent, therefore for this application an uninterruptible power source (UPS) is highly recommended. Even this may not be adequate, if the voltage drops below 80 V which is the control limit for most UPS units. Experiments were made in which the line voltage was decreased in 10 V intervals from 110 V to 10 V and then suddenly increased to 110 V. The output decreased rapidly as the voltage decreased. After the level was quickly raised from 10 to 110 V the sensor no longer functioned properly. Thus, for stable, repeatable operation the instrument power should not be allowed to vary more than ±2 percent. To verify that no changes in sensitivity have occurred, a reference surface should be measured periodically. The reference used by MSFC is vapor deposited chrome with a thickness of 2000 to 3000 Å. If properly prepared, a smooth homogeneous surface is obtained. The reference is kept covered and handled only at the edges with gloves.

CALIBRATION METHOD AND RESULTS FOR SRM CASE INSPECTION

The proper calibration of the sensor for each specific measurement application is necessary if quantitative contamination measurements are to be made. The procedure used and the result obtained for the inspection of the SRM D6AC steel case are presented below.

The SRM case is made of D6AC steel which is subject to corrosion. A corrosion inhibiting grease, Conoco HD-2, is applied to protect the surface during shipment and storage. Prior to the processing of the case segments for the bonding of the rubber insulation to the steel, the case cylinders (12-ft diameter by 13.5-ft long) are degreased using a methyl chloroform vapor degreaser. The cylinders are then inspected using a "black light" to verify that the grease has been removed by the degreasing process. Following the "black light" inspection, the O-ring grooves are carefully greased and two case cylinders are mated to form a casting segment that is approximately 12 ft in diameter and 27 ft long. The segment is then moved to a pit where the exterior is painted and the Chemlock adhesive system is applied on the interior. Green rubber is then laid on the adhesive and the segment is placed in an autoclave for vulcanization at 100 psi and 300°F for 3 hours. The requirement for the strength of the rubber-to-case bond is 150 lb/in. (PLI) in 180-deg peel. Because of the desire to enhance the strength and reliability of this critical bond, an investigation was initiated to determine the case cleanliness level necessary to repeatedly achieve the 150 PLI requirement. In addition, the effectiveness of the "black light" inspection in detecting case contamination was to be established.

To establish the required cleanliness level, peel strength versus contaminant level tests were made. One-foot by one-foot steel plates were cut from a scrap case cylinder so that surfaces identical to the case were used for the tests. The surface roughness as measured was 100 μin. The plates were grit blasted and then remeasured to verify that the grit blasting had not changed the finish. After grit blasting, they were methyl chloroform vapor degreased and then ultrasonically cleaned in a methyl chloroform bath. The cleanliness level of each plate was then determined by NVR analysis to be less than 0.25 mg/ft^2. The plates were contaminated by spray application of Conoco HD-2/methyl chloroform solutions to provide nominal grease levels of 1, 5, 10, 50, 100, 200, 500, and 1000 mg/ft^2. The levels were checked by spraying 1-ft by 1-ft aluminum foil and weighing to verify the quantity of HD-2 applied to the plates. OSEE measurements were made on clean plates and those that had been contaminated. The excellent correlation established between the OSEE sensor response and the nominal contaminant level up to 50 mg/ft^2 is shown in Figure 10. A gain change from 5 to 6 increased the response but did not change the slope. OSEE data for 100 mg/ft^2 and higher levels are not shown since the HD-2 grease is photoemitting. Beyond 50 mg/ft^2 the OSEE response dropped significantly and then increased. This was first due to the complete absorption of the photoelectrons from the steel, then the total absorption of the UV in the grease, and finally the subsequent increased photocurrent from the grease.

Upon completing the OSEE measurements, the Chemlock primer and adhesive and the NBR rubber were applied exactly according to the SRM process. The plates were vacuum bagged and autoclaved. Upon completion of processing, eight 1-in. wide strips were cut in the rubber on each plate and 180-deg peel tests were made. The test results are graphically depicted in Figure 11. No bonding was achieved for HD-2 contaminant levels of 1000 and 500 mg/ft^2 (Fig. 12). Only 5 PLI average strength was obtained at 200 mg/ft^2 and failure was 100 percent adhesive (Fig. 13). At 100 mg/ft^2 a peel strength of 59 PLI was attained and the failure was 60 to 80 percent adhesive (Fig. 14). For contaminant levels of 50 mg/ft^2 or less, the failure was cohesive in the rubber (Figs. 15, 16, and 17). However, the peal strength continued to increase significantly as the contaminant level decreased. Based on this observation, it was postulated that the HD-2, which has a high vapor pressure, was diffusing through the Chemlock during the vulcanization process (autoclaving at 100 psi, 300°F, 3 hr) and degrading the rubber. Subsequent tests showed that the grease experienced an 80 percent weight loss under these conditions and residual gas analyzer tests verified that HD-2 would diffuse through Chemlock at 300°F. Additional tests determined the effect on NBR rubber properties due to HD-2 diffusion into the rubber. The test specimens and data are shown in Figure 18. As can be seen, the diffused grease plasticized the rubber resulting in a decrease in tensile strength from 1234 psi to 935 psi and an increase in elongation from 87 to 522 percent. These data substantiated the hypothesis that small quantities of diffused HD-2 could significantly affect the post-vulcanization mechanical properties of the NBR rubber. It was concluded from these tests that the HD-2 contaminant level must be controlled to a level of 5 mg/ft^2 or less if a composite bond line peel strength of 150 PLI was to be achieved repeatedly and that a level of 50 mg/ft^2 or less was necessary to assure no failure in the adhesive. It was also demonstrated that the OSEE technique could be used to measure HD-2 levels on D6AC steel over the range of 0.25 to 50 mg/ft^2.

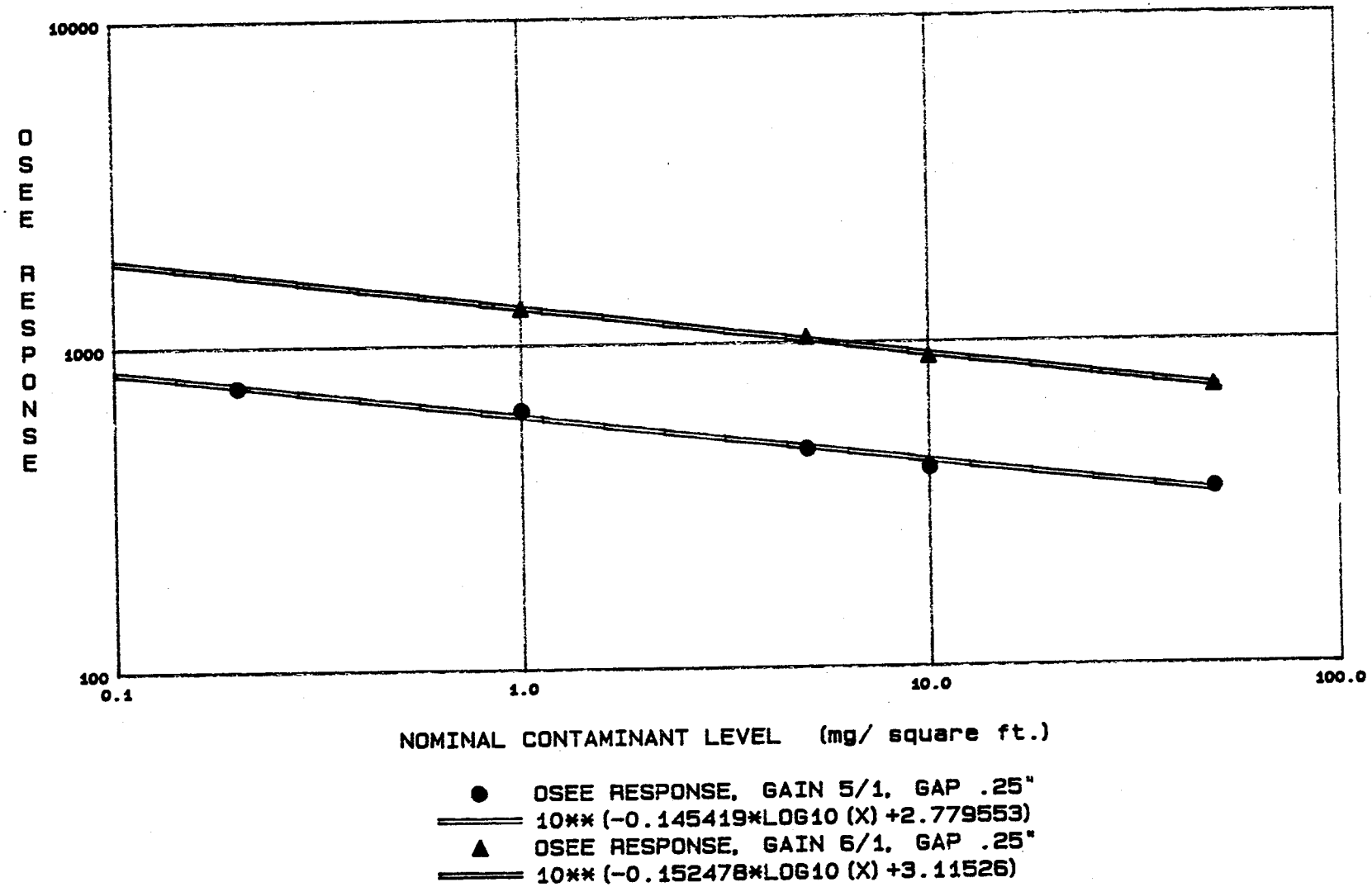

Figure 10. OSEE response versus HD-2 contaminant level (D6AC steel).

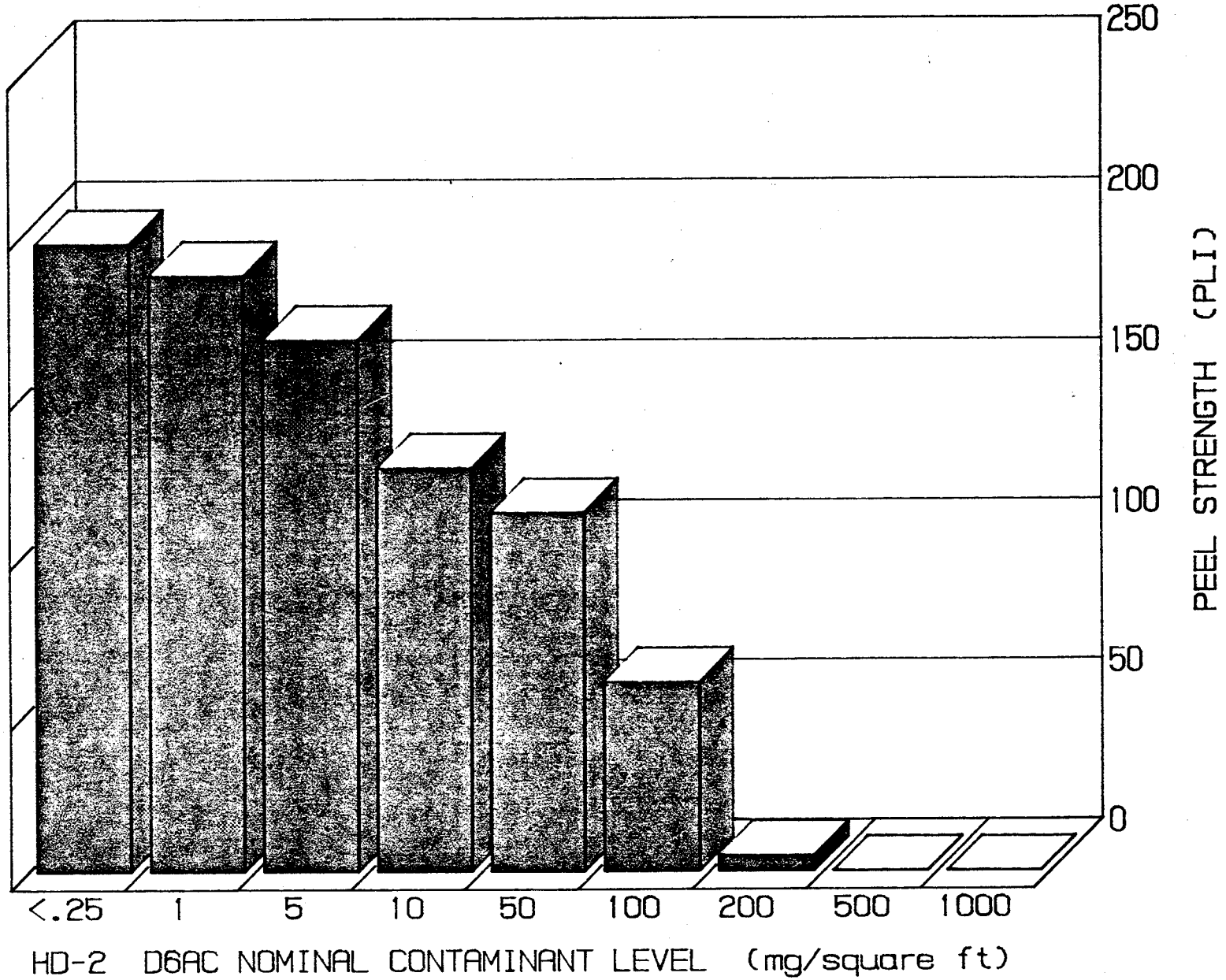

Figure 11. Effect of HD-2 contaminant on NBR peel strength.

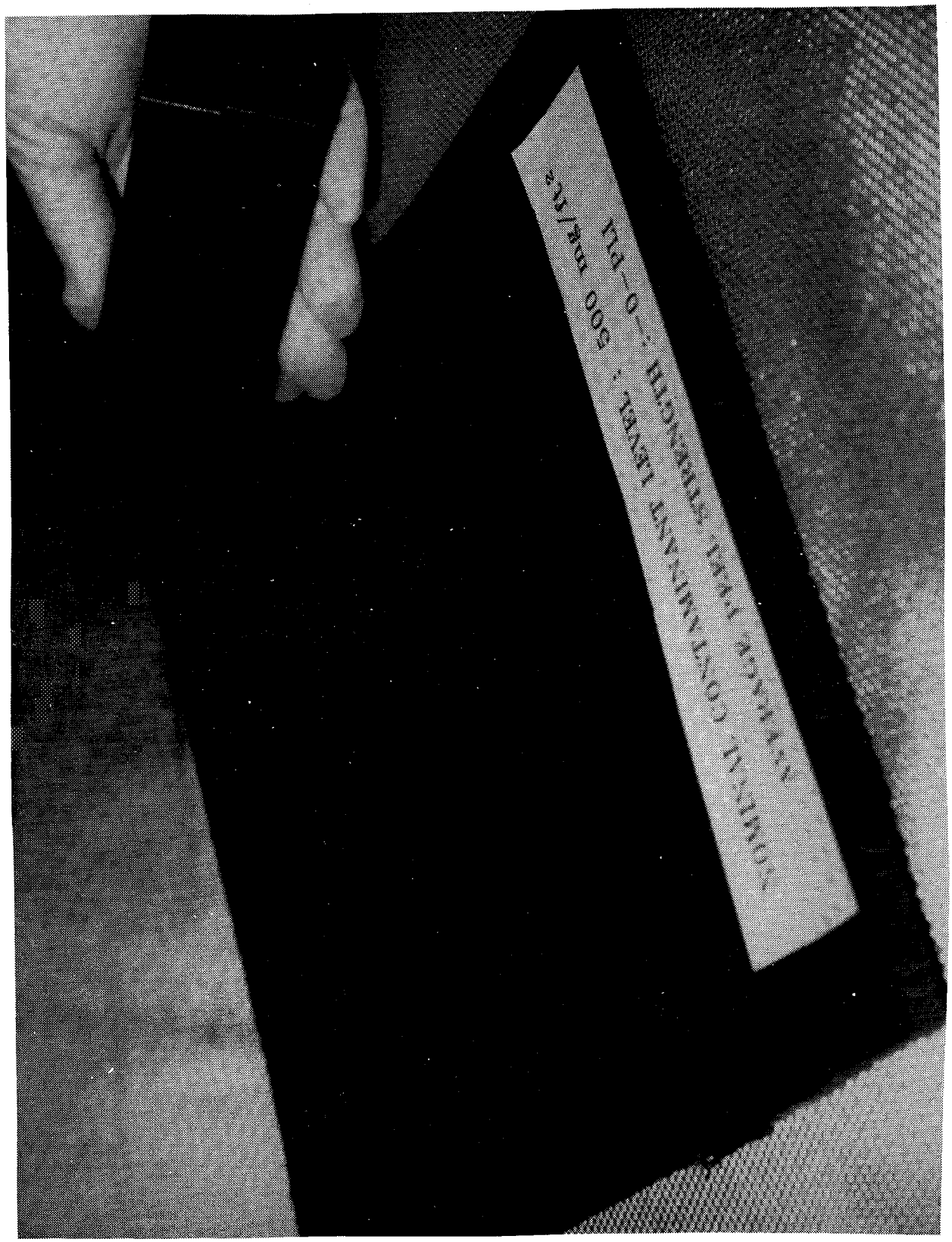

Figure 12. 500 mg/ft^2 peel strength specimen.

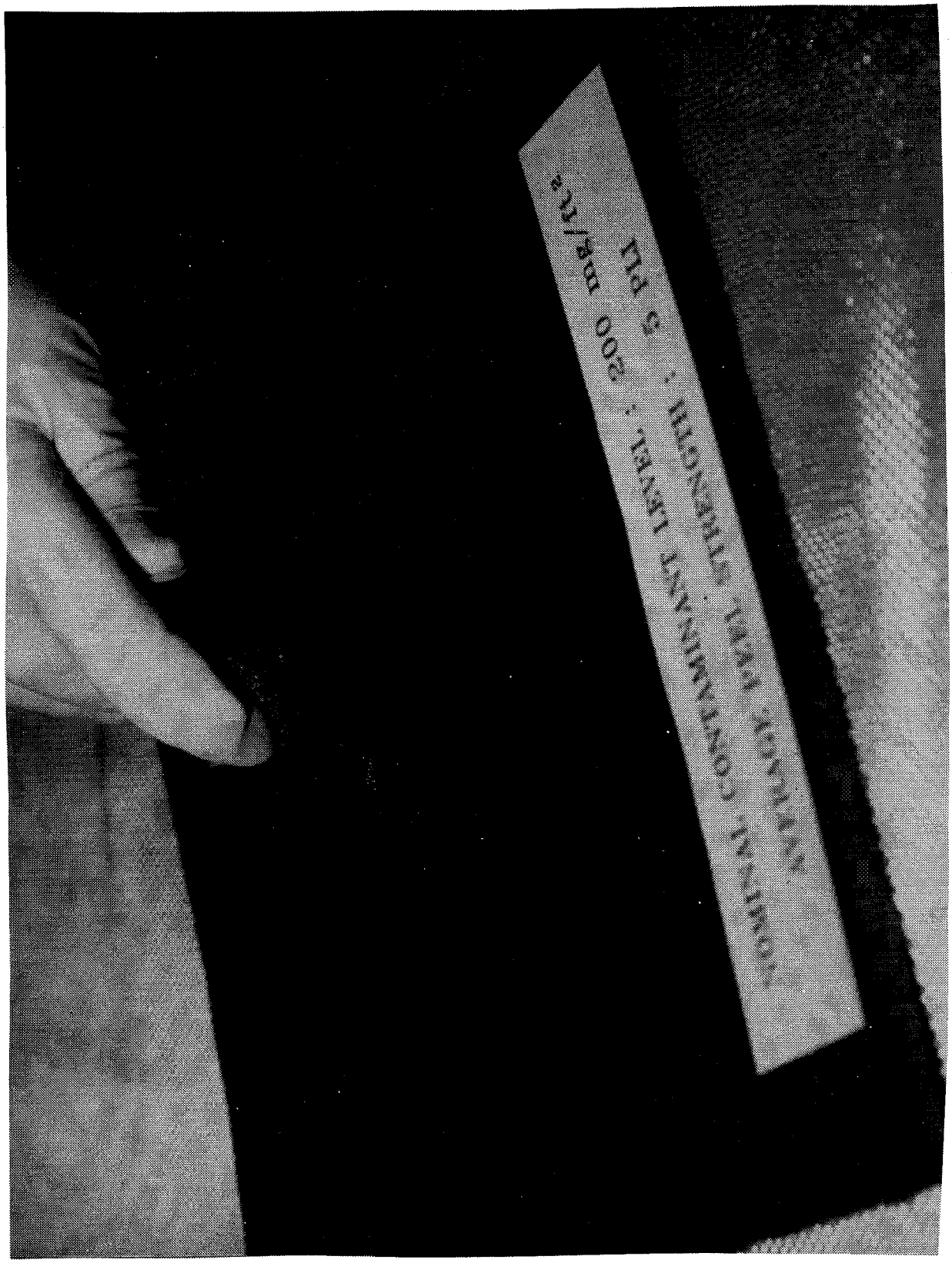

Figure 13. 200 mg/ft^2 peel strength specimen.

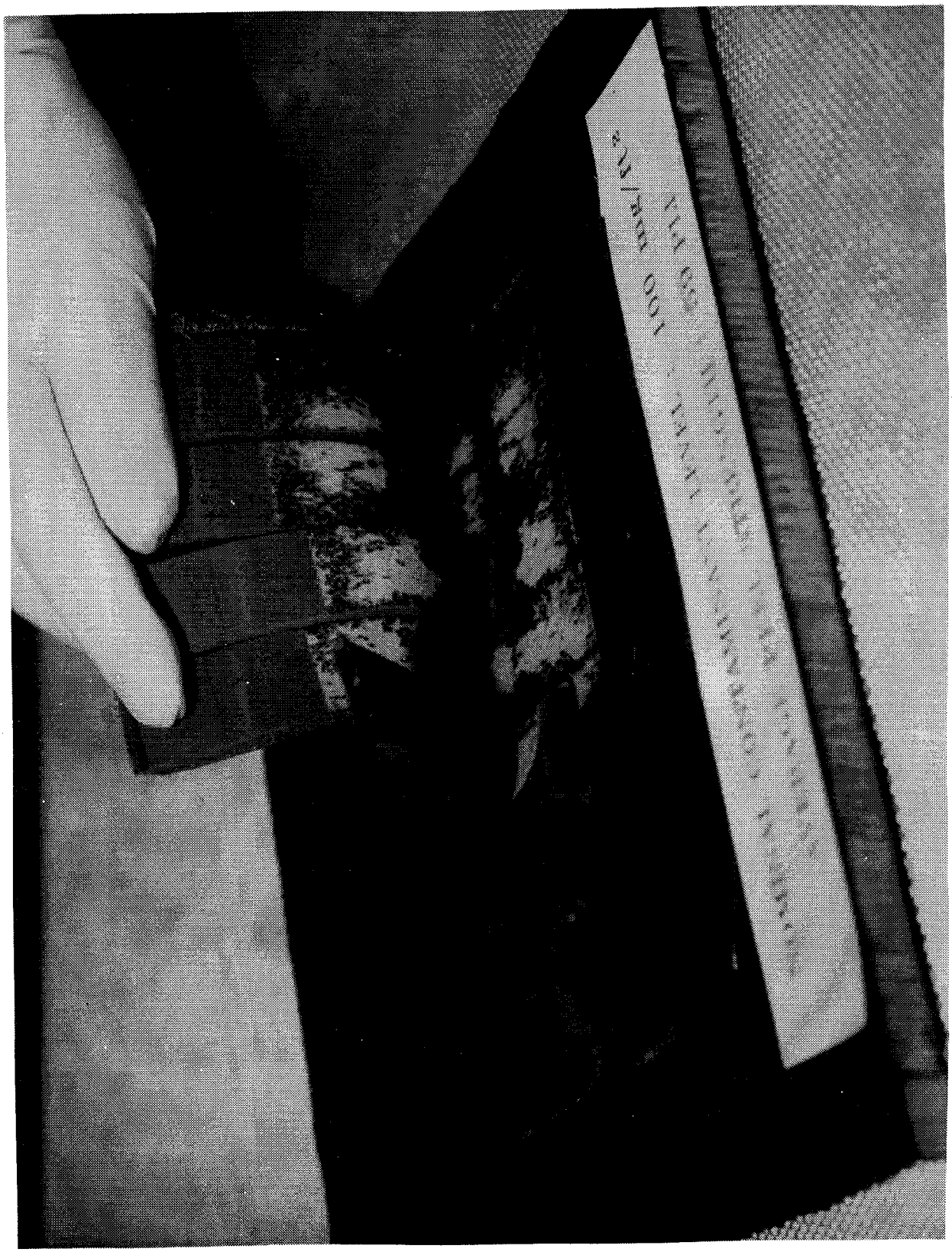

Figure 14. 100 mg/ft^2 peel strength specimen.

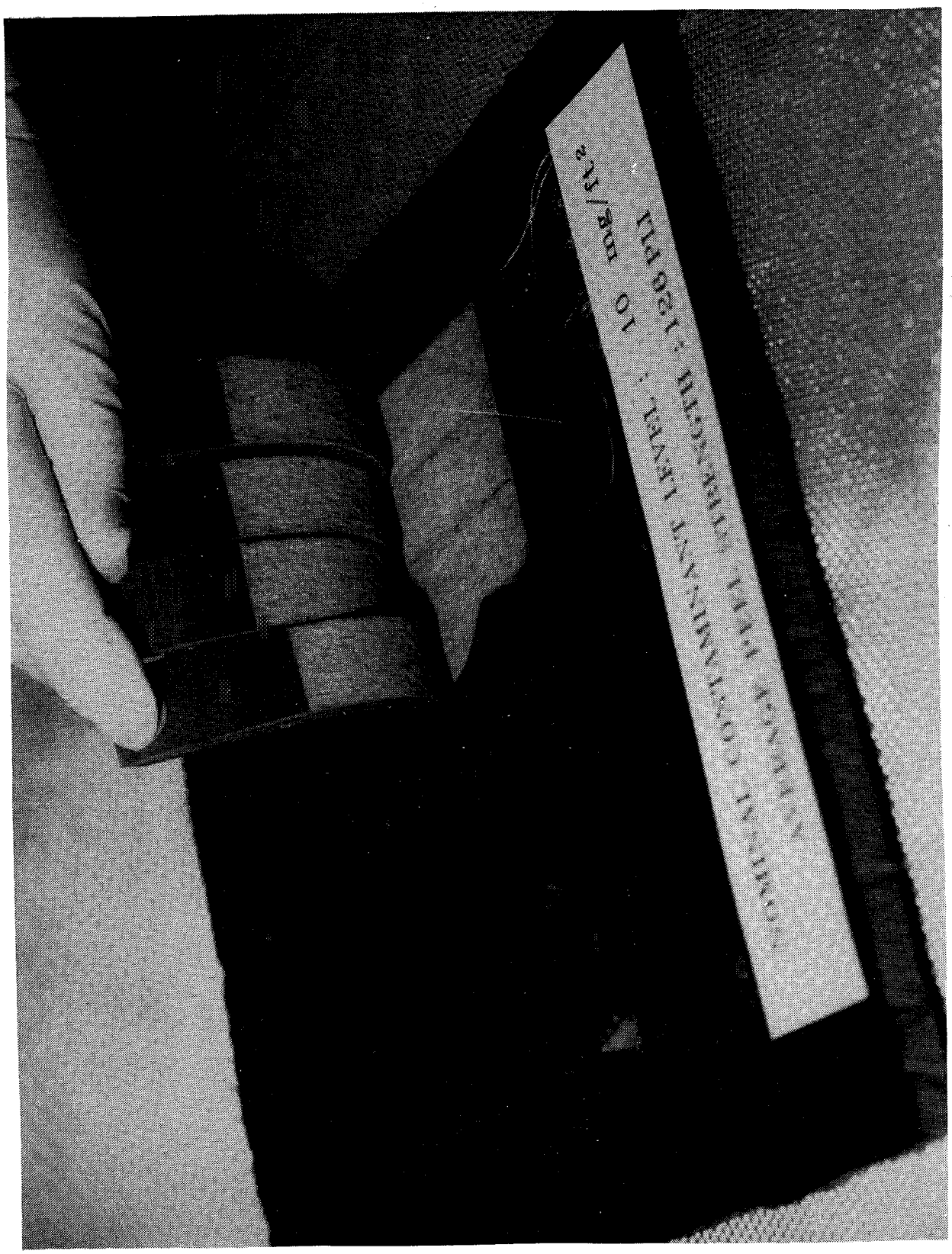

Figure 15. 10 mg/ft^2 peel strength specimen.

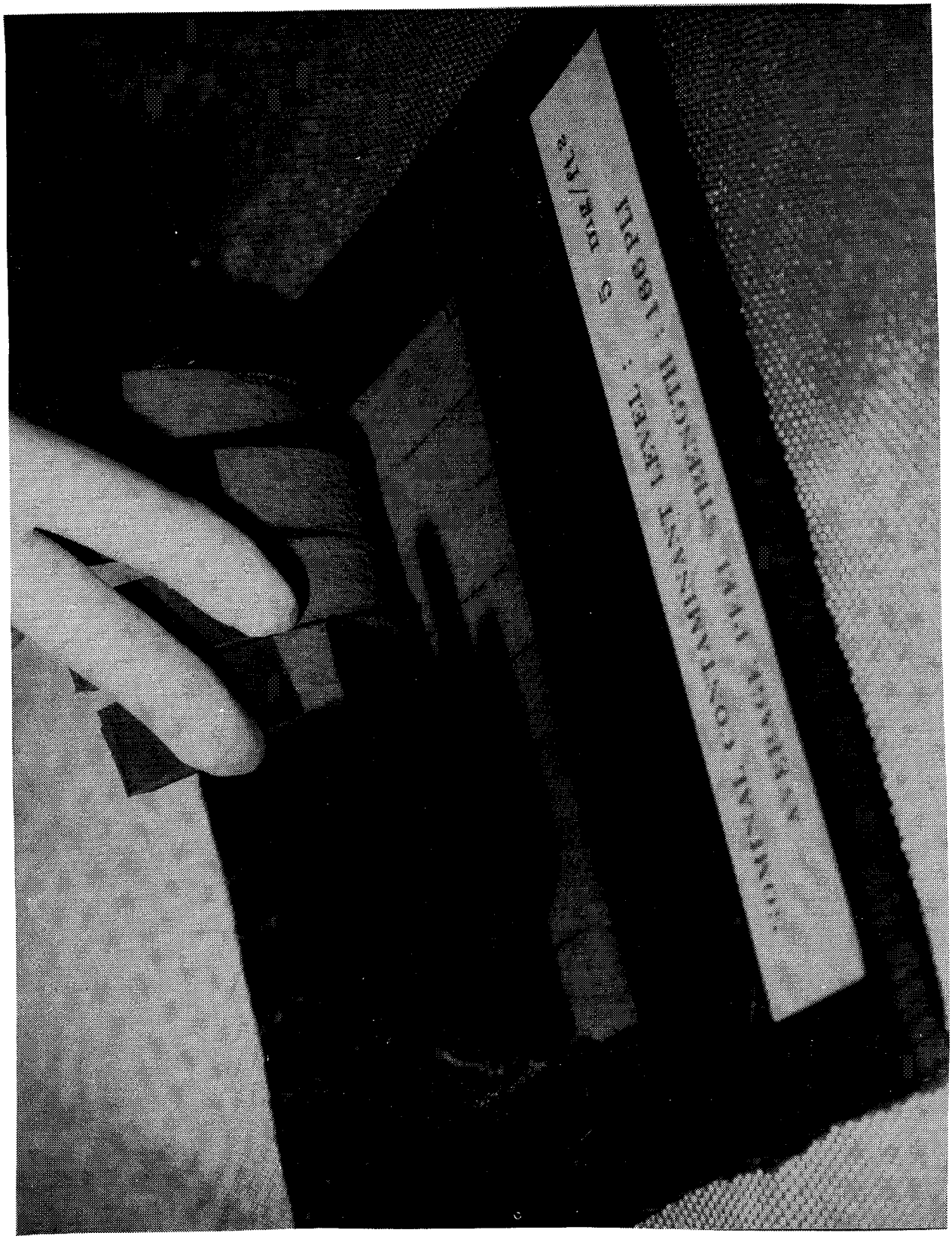

Figure 16. 5 mg/ft^2 peel strength specimen.

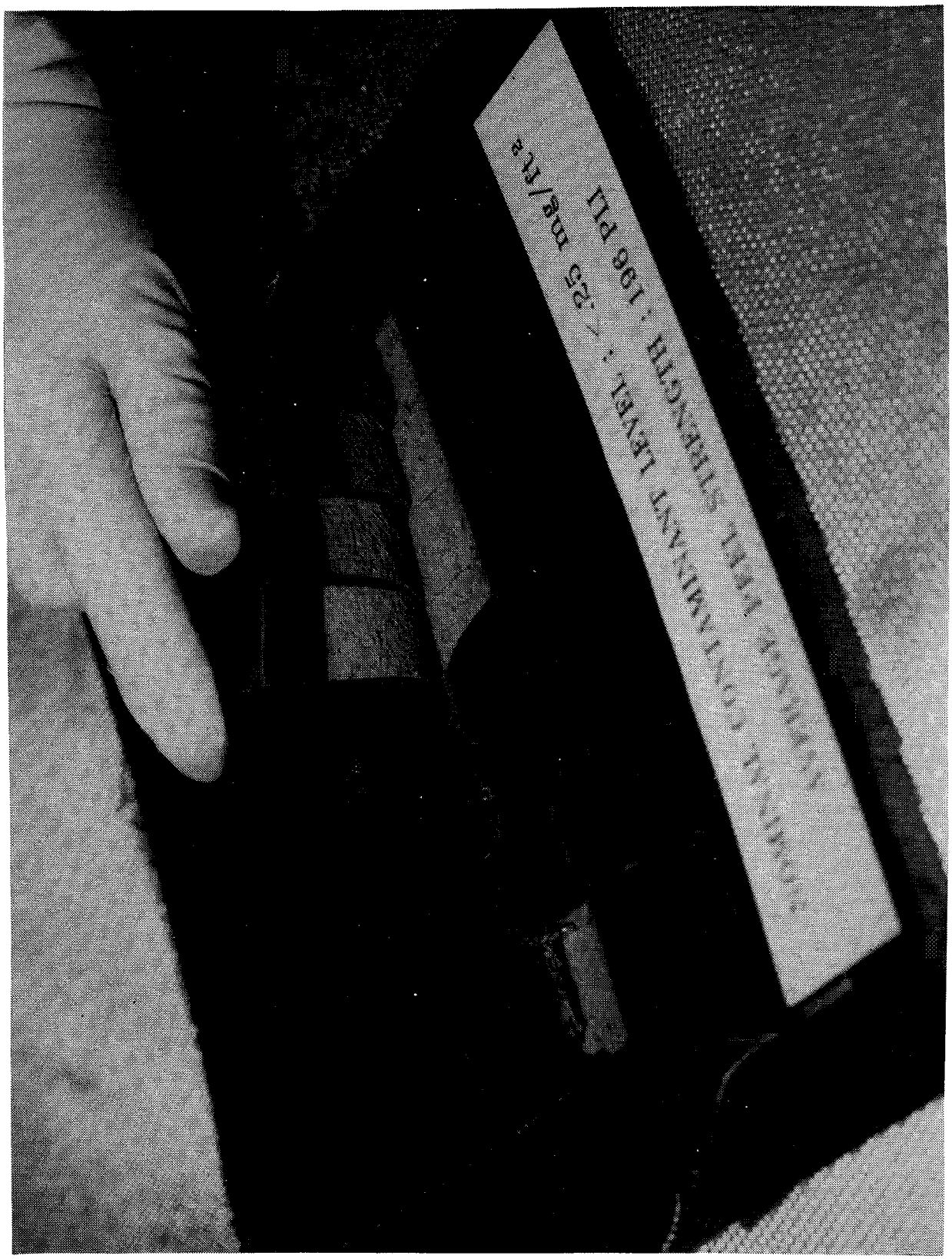

Figure 17. <0.25 mg/ft^2 peel strength specimen.

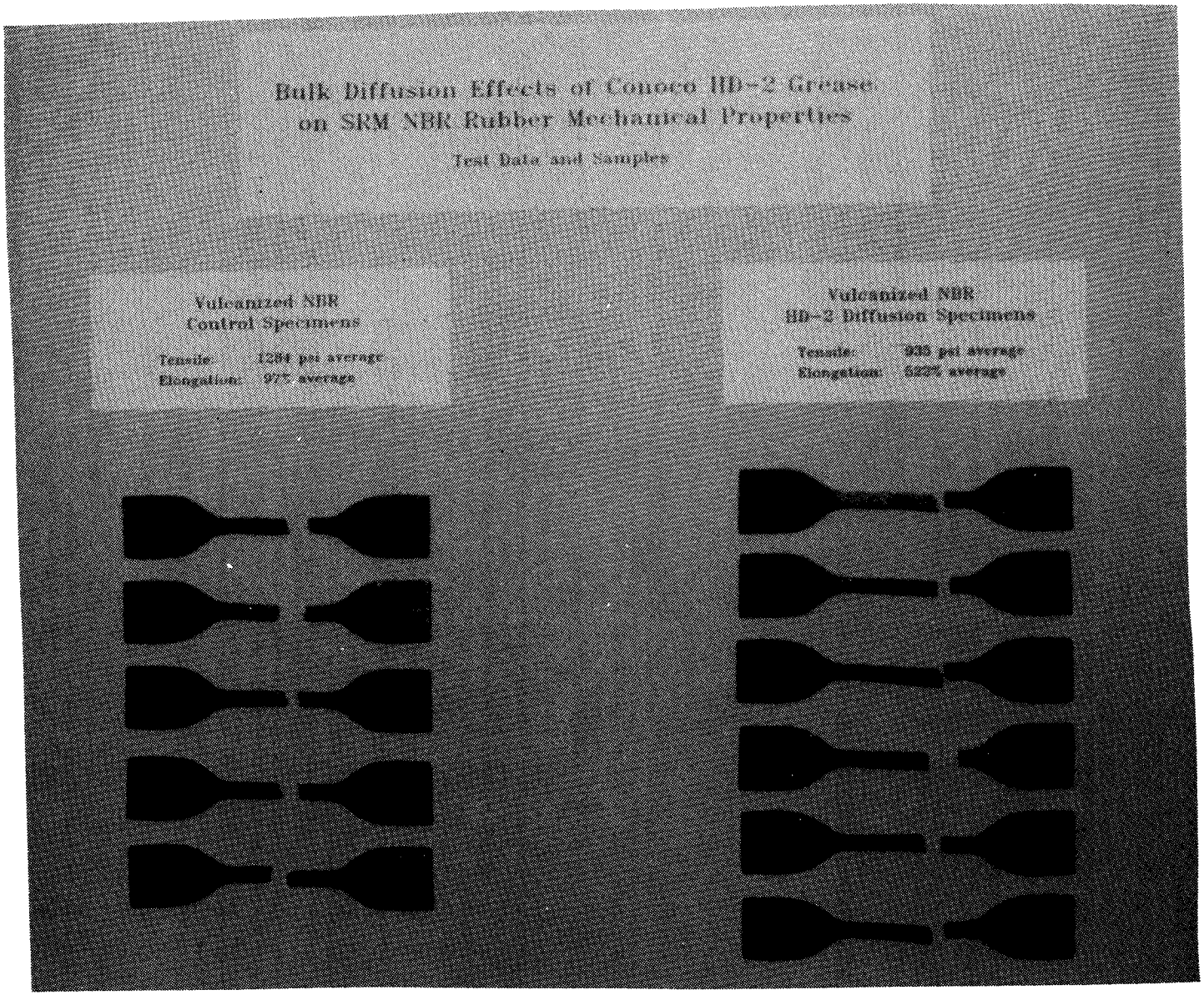

Figure 18. Bulk diffusion effects on Conoco HD-2 grease on SRM NBR rubber mechanical properties.

As stated previously, "black light" inspection sensitivity was to be determined during these tests. Under optimum observing conditions, a uniform contaminant level of 100 mg/ft^2 or greater could be detected. Thus, the black light and the OSEE technique complement each other. The "black light" is useful for detecting gross contaminent levels but cannot detect the levels necessary to achieve quality bonding. The OSEE technique can detect these low levels but cannot be used for the high levels. Thus, the case should first pass a "black light" inspection and then be inspected with the OSEE technique for verification of cleanliness requirements prior to bonding.

Figure 19 graphically illustrates how the combination of surface finish and contaminant level influenced the peel strength results discussed above. In the figure, the contaminant level is overlaid to scale on a surface profile of the D6AC steel plate. The measured surface finish is 100 μin. or 25400 Å. Since the density of HD-2 grease is very near 1 gm/cm^3, a contaminant level of 1 mg/ft^2 is equivalent to a uniform areal thickness of ∿108 Å. Thus, a level of 250 mg/ft^2 correlates to a thickness of 27000 Å or roughly 106 μin. This quantity of grease then would be sufficiently thick to completely cover the surface thereby preventing any adhesion between the Chemlock and the steel. Thus, it is not surprising that there was zero peel strength at levels greater than 250 mg/ft^2. As the contaminant thickness decreases, the peel strength increases as more steel bonding surface is exposed. Sufficient asperity area is available for good ahesion at the 50 mg/ft^2 since this is where the failure mode transitions from adhesive to cohesive (failure in the rubber). The grease trapped in the valleys then becomes important because the autoclaving temperature and time conditions cause it to diffuse through the Chemlock into the rubber resulting in a degradation of the rubber near the Chemlock-rubber interface. As the quantity of available grease decreases, the degree of degradation is less and the strength is greater. If the surface were smoother, less grease would be trapped and cleaning would be easier. Conversely, the effective area for adhesive bonding would be decreased since the projected area of the rough surface is greater than that of a smoother surface.

SRM CASE INSPECTION DEMONSTRATION

With the establishment of the SRM case cleanliness requirements for HD-2 grease (the principal case contaminant) and the calibration of the OSEE technique for verifying the requirements, a full scale demonstration of the prototype ConScan system described earlier was performed at the MTI SRM manufacturing facility in Utah. Figure 20 shows the installation of the sensor and associated position controller on the hydraulically operated cage and the location of the sensor relative to the casting segment which was mounted on a turntable. The data system is shown in Figure 21.

Scans were made at a rate of 30 ft/min of a nonflight casting segment after it had been vapor degreased and stored uncovered in the manufacturing area for several days to simulate the flow of a flight segment. A scan of a portion of the as-received segment is shown in Figure 22. The dark areas are where the contaminant level was greater than 5 mg/ft^2. After this inspection was made, several areas scanned previously were cleaned using two different methods. One method involved hand cleaning with a wipe saturated with methyl chloroform followed by a dry wipe. The other

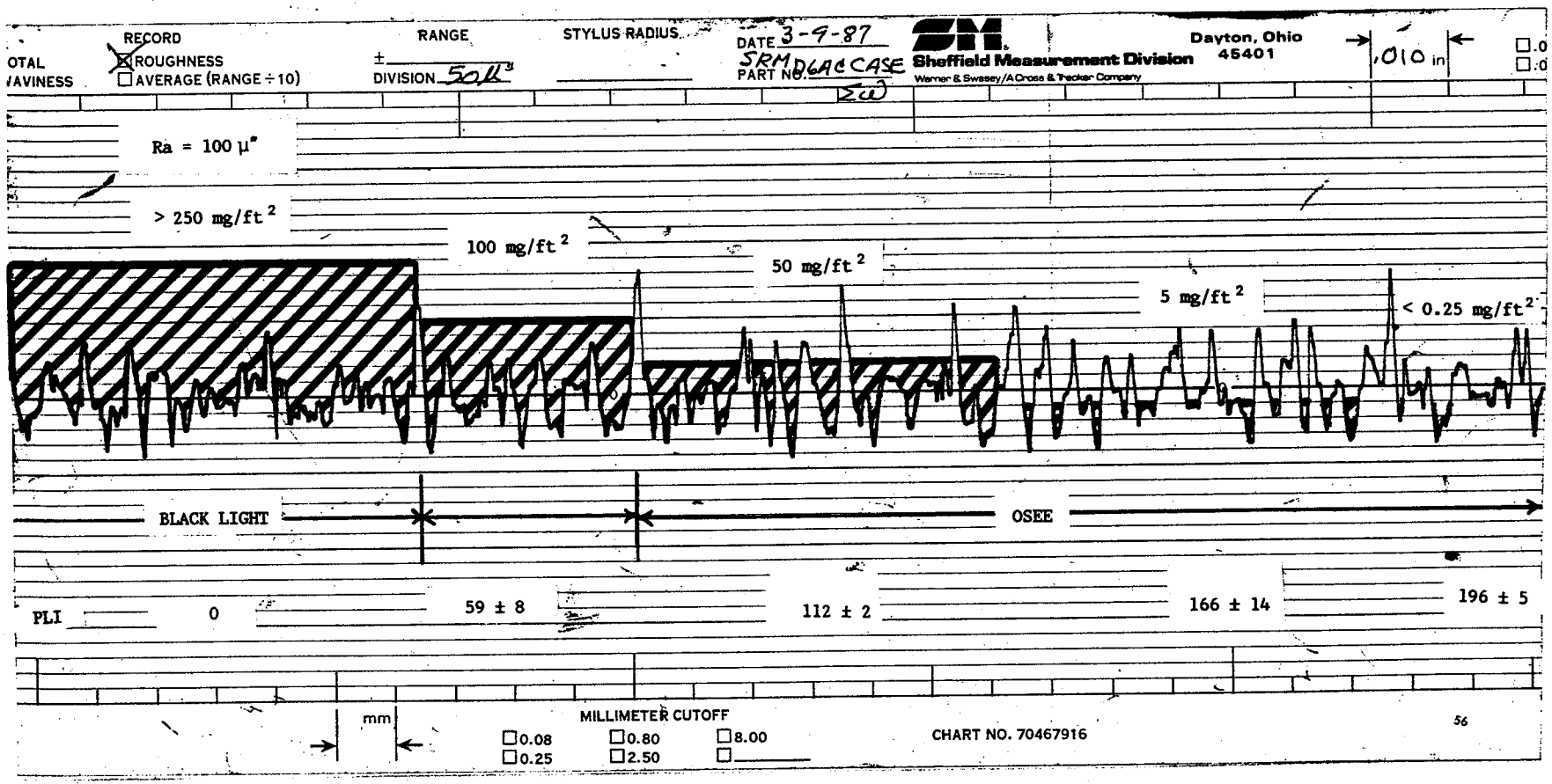

Figure 19. Surface finish and contaminant levels versus peel strength.

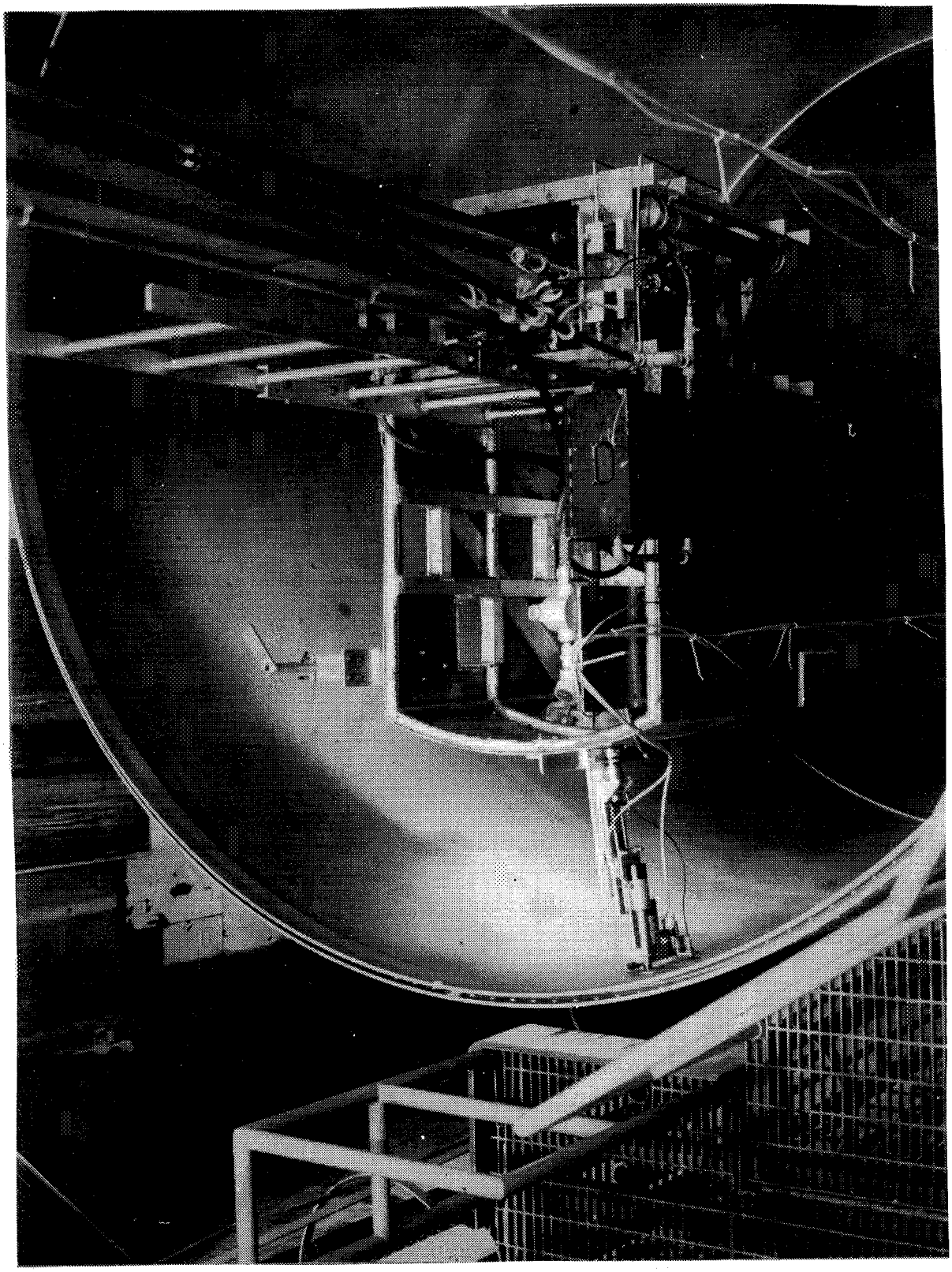

Figure 20. ConScan system SRM demonstration inspection using PAT OSEE sensor.

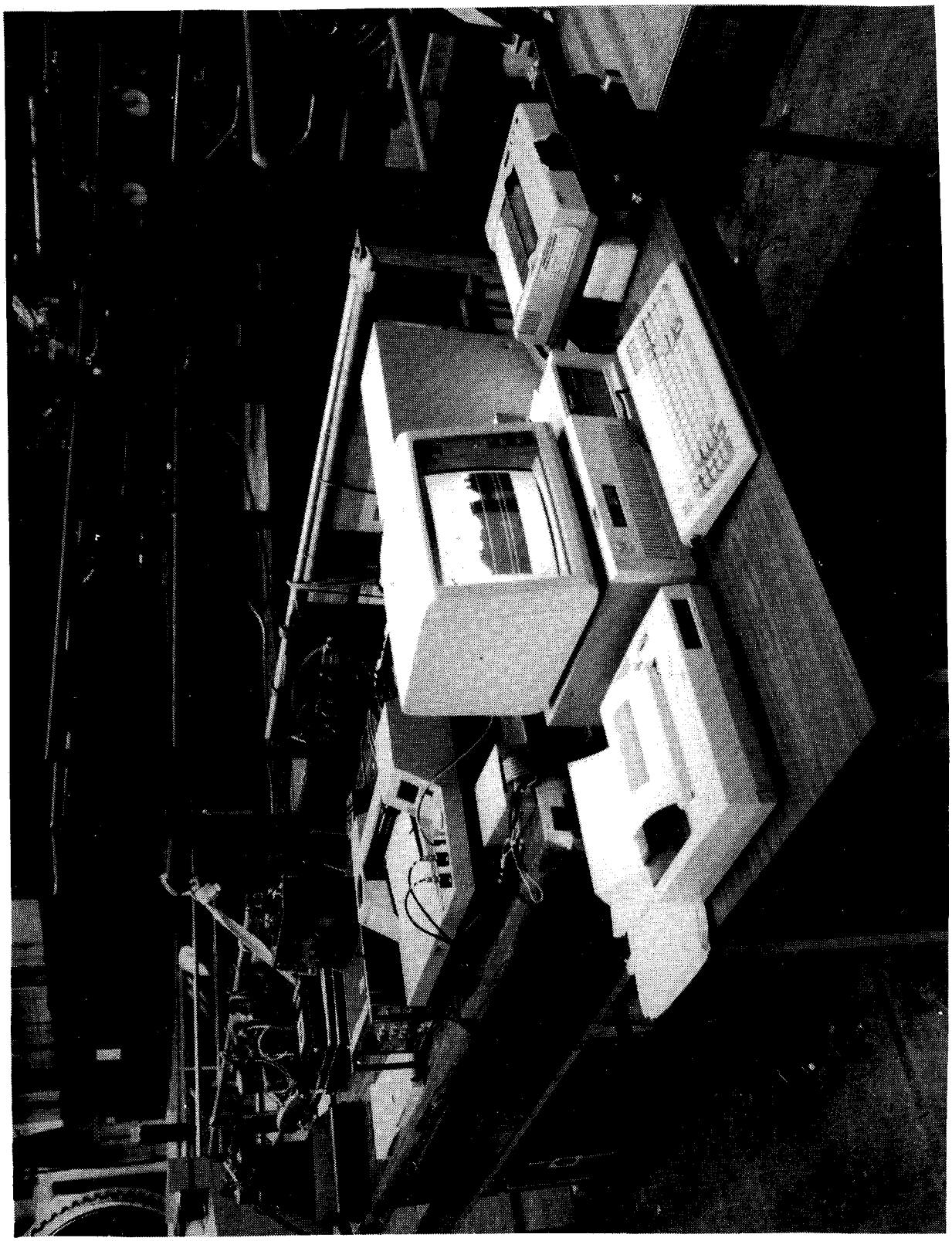

Figure 21. ConScan data system.

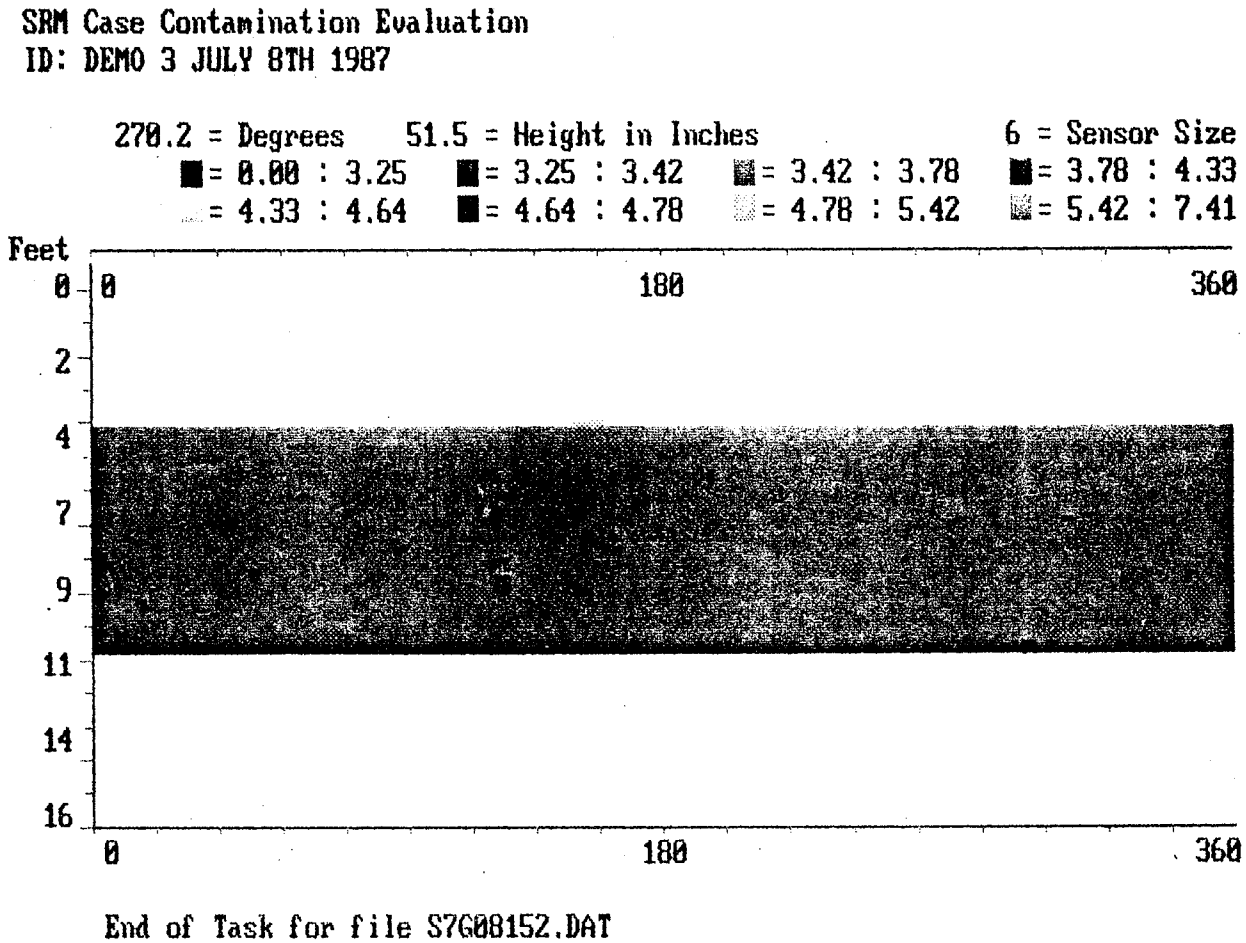

Figure 22. Scan of as-received segment using ConScan.

method utilized a pressurized container with a fan-shaped nozzle filled with methyl chloroform for flushing the surface. The result of these cleaning operations is presented in Figure 23. The lightest (cleanest) areas were where the flushing method was used. Hand cleaning was not as effective due to the roughness of the surface.

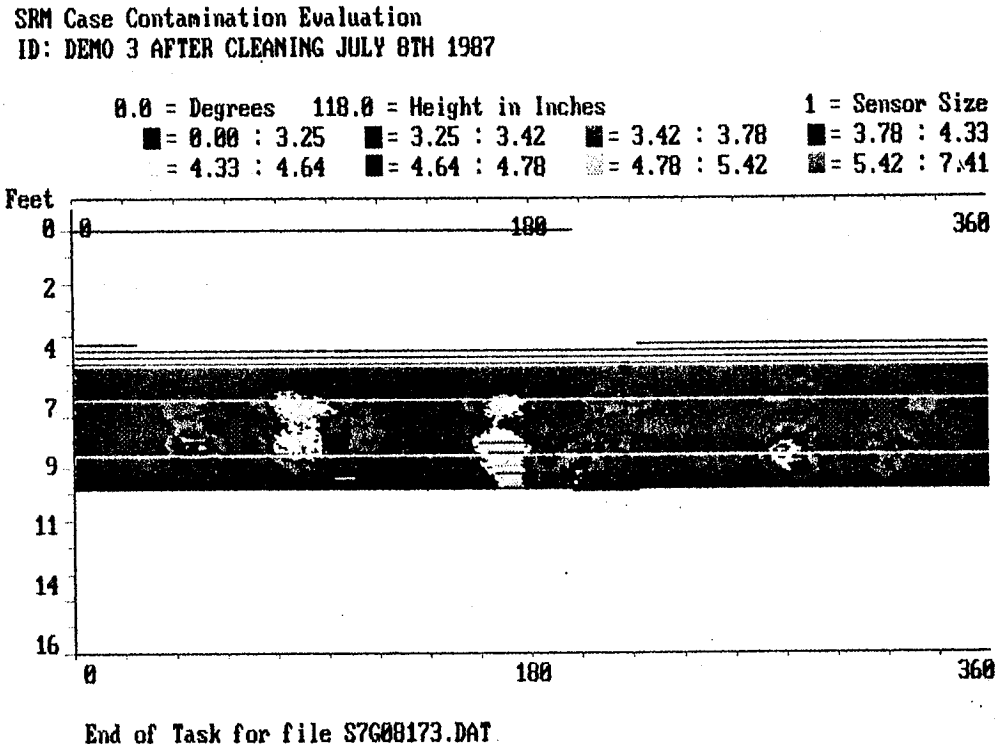

Figure 23. Scan of segment after cleaning.

Sufficient scans were made to demonstrate the feasibility of the technique for the inspection of SRM flight segments. The design of an on-line system for routine SRM inspection has been initiated based on the prototype used for the demonstration.

OTHER OSEE APPLICATIONS BEING INVESTIGATED

Because of the successful results achieved for the SRM Case bond enhancement effort, other applications for the OSEE technique currently are being investigated. These include Space Shuttle SRM Nozzle, SRB Booster Separation Motor, and External Tank bonding surface inspection. A brief description of the status of each is given in the following.

SRM Nozzle

The nozzle is comprised of many large bonded parts. Both metal-composite and composite-composite bondlines are utilized. Also, there is a wide range of potential contaminants including fingerprints, oils, greases, release agents, and silicones which could degrade bond quality. Laboratory testing is in progress to characterize

the effects of these contaminants on bond strength and to develop OSEE response and calibration data for the various surfaces and contaminants.

The MSFC OSEE laboratory system used to obtain these data is shown in Figure 24. An OSEE sensor is used to scan test specimens mounted on a computer-controlled X-Y table. The desired sensor-to-specimen spacing is established using a Z-axis translation controller. The area to be scanned and the number and spacing of the measurement points are software selectable. At the completion of a scan, the data are displayed in a 3-D format. The total system, including the computer, I/O boards, and special software, is commercially available from PAT. Figure 25 is a scan made of a 7075-T73 aluminum specimen that had been dry sanded with 230 grit abrasive, wiped with methyl chloroform, and then contaminated with a fingerprint. By subtracting the original non-fingerprinted scan from that shown in Figure 25, the contamination from the fingerprint is clearly shown (Fig. 26). The surface was sequentially wiped with methyl chloroform, toluene, and then methyl ethyl ketone (MEK). The results are shown in Figures 27, 28, and 29. Wiping did not appear to be an effective means of removing a fingerprint, particularly with methyl chloroform since all it did was smear the contaminant over a larger area.

Since contaminants from sources other than fingerprints are of concern, preliminary tests were made to determine the capability of the OSEE sensor to detect these on one of the nozzle bonding surfaces. Since EA 9228 epoxy primer is being investigated for improving the bonding between 7075-T73 and its mating surface (glass phenolic), several different contaminants at low levels (on the order of 2 to 5 mg/ft^2) were applied on the material surface. Figure 30 is a scan of the original surface containing an area near the edge with an unknown contaminant. Figure 31 shows the effect of various contaminants and Figure 32 illustrates the result of wiping with methyl chloroform. The effectiveness of flushing the surface is dramatically demonstrated by the scan shown in Figure 33.

Since the OSEE technique has the capability, as shown by these tests, to detect the contaminants of interest, efforts are now underway to develop specific calibration data for these contaminants on the various nozzle bonding surfaces.

Some measurements on full-scale SRM nozzle parts have been made. However, due to the present lack of an automated scanning capability, only spot inspections were made. The use of the OSEE sensor on an SRM nose inlet housing is shown in Figure 34. In this application, a clean standoff with a non-photoemitting coating is used to maintain the sensor-to-surface spacing. Since spot inspections by hand are time consuming and the probability of missing a contaminated area is high, automated scanning of the total bonding surface is highly desirable. Figures 35 and 36 show a gantry robot and the robot arm with an OSEE sensor being used to scan a 10-ft diameter test article mounted on a turntable. This system is under development for the automated scanning of the large SRM nozzle parts.

BSM Investigations

Figure 37 shows the development setup for scanning of the BSM Case which is 7075-T73 aluminum. The ConScan system described earlier is being used for this application. Representative scans of the BSM Case are shown in Figures 38 and 39. The first scan shows the case prior to cleaning. The second scan shows the effect of sanding throughly. The dark spots are very thin layer contaminants (release agent, top and hydraulic oil, bottom) that were applied on the otherwise clean area.

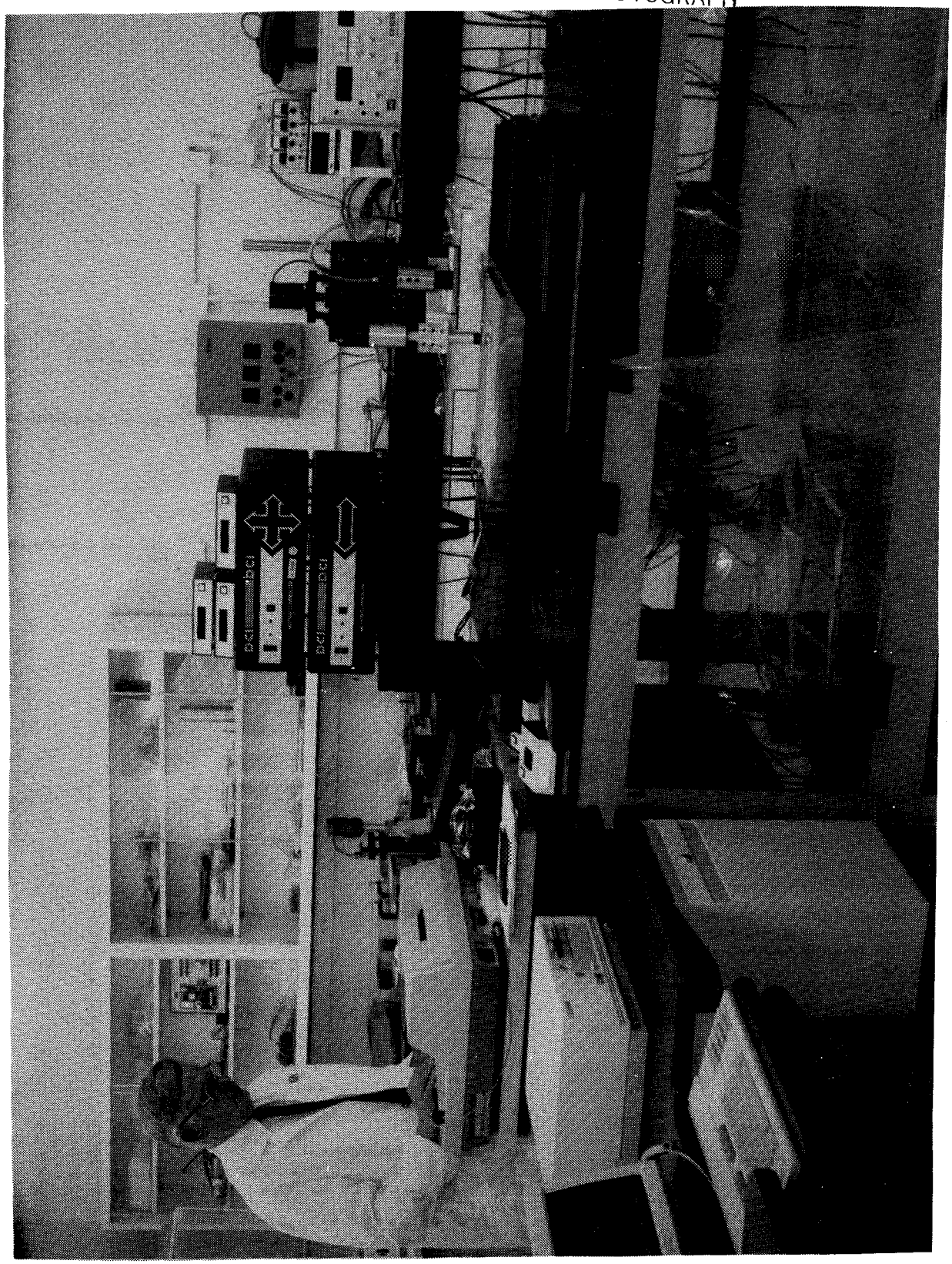

Figure 24. MSFC Laboratory OSEE PatScan system.

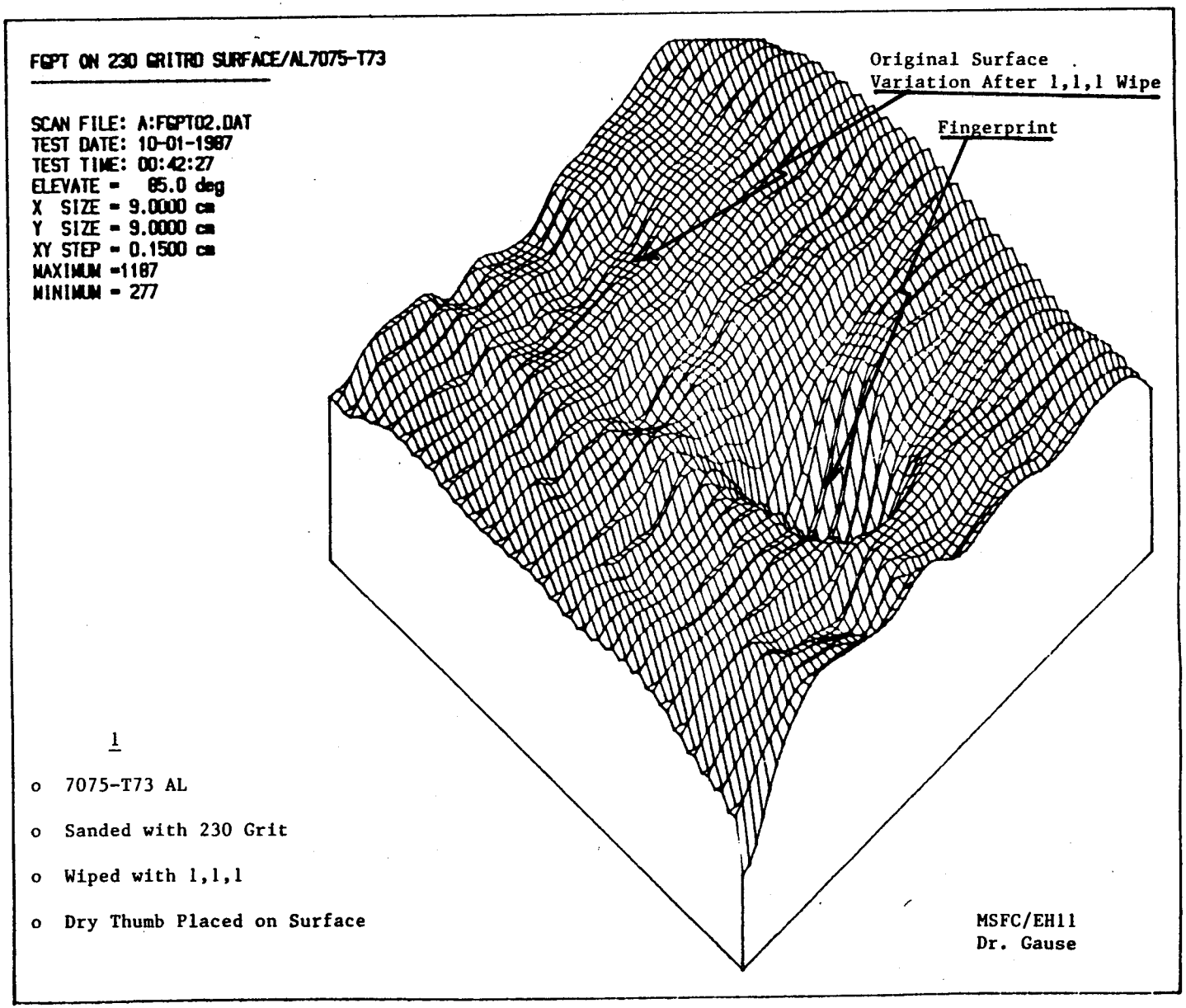

Figure 25. OSEE scan of fingerprint on 7075-T73.

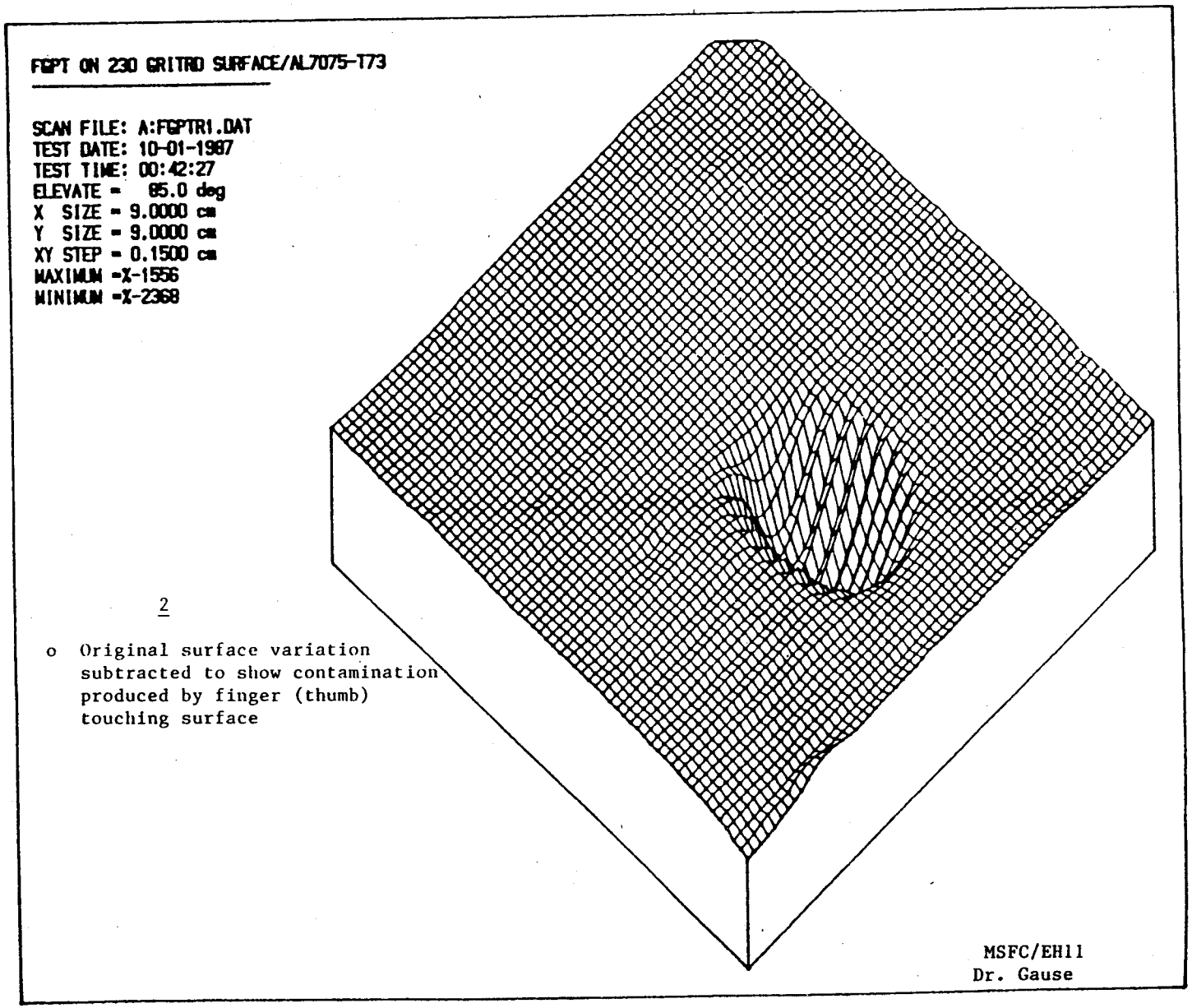

Figure 26. Contamination from fingerprint.

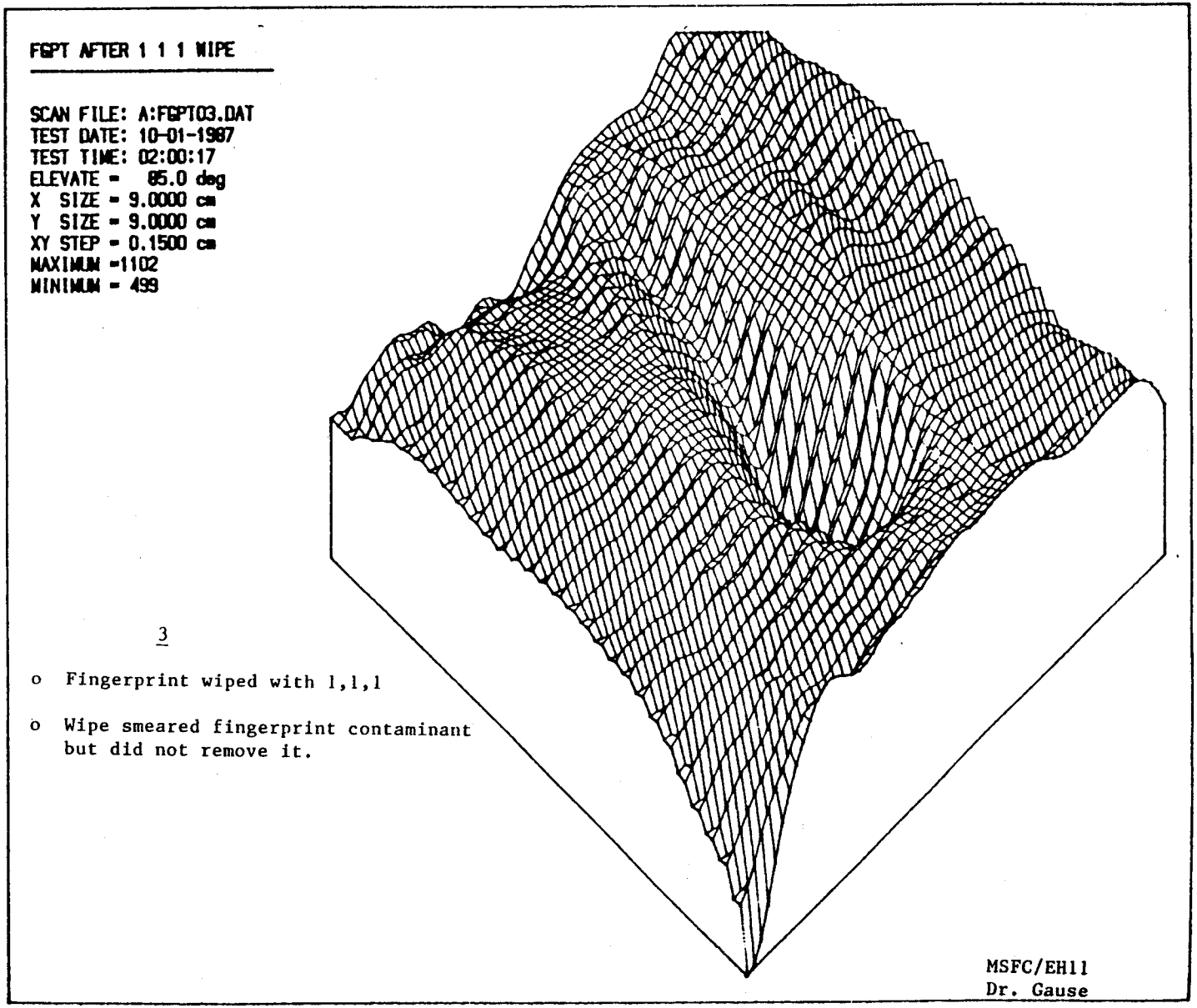

Figure 27. Fingerprint after methyl chloroform wipe.

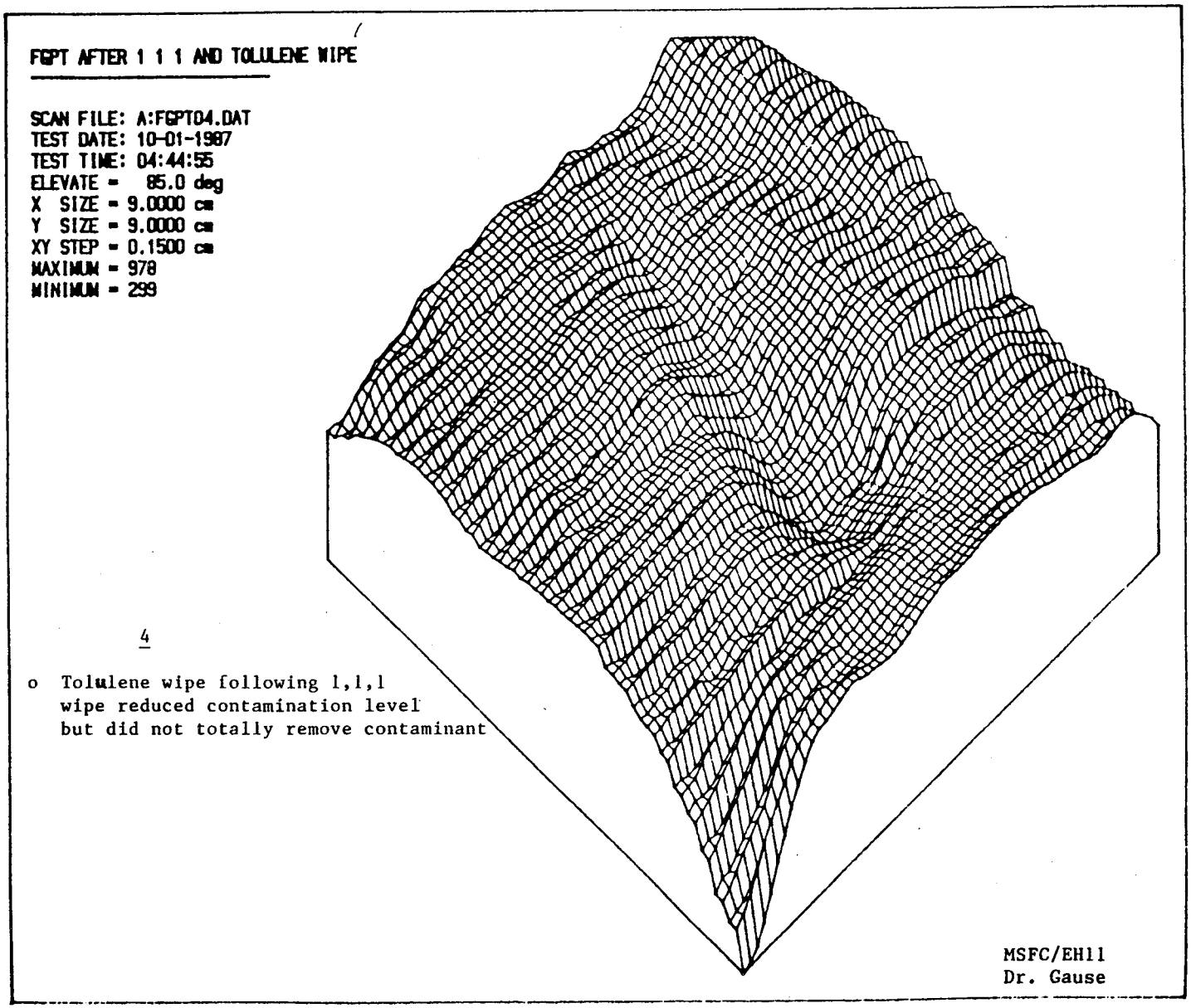

Figure 28. Fingerprint after wiping with methyl chloroform and toluene.

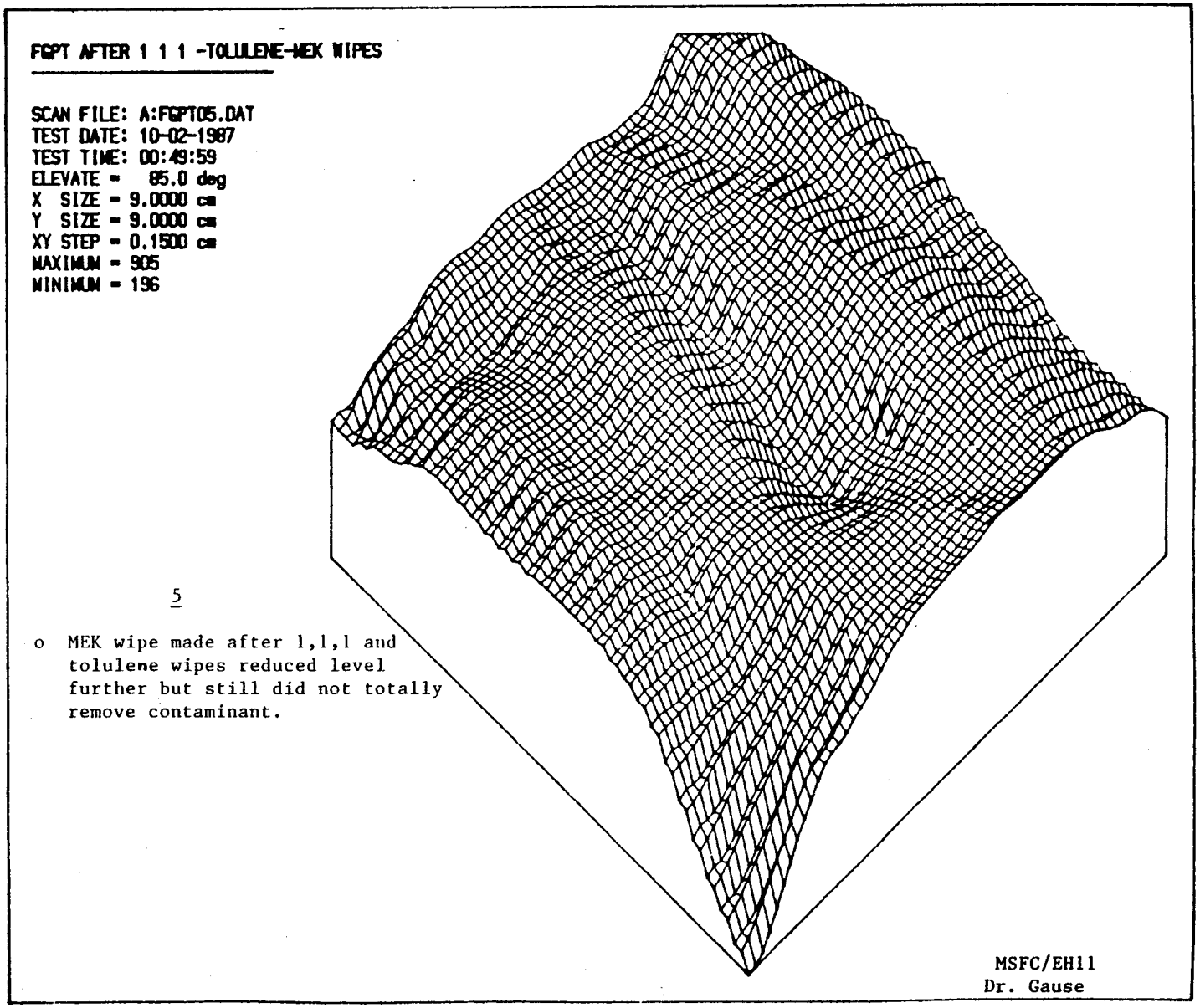

Figure 29. Fingerprint after wiping with methyl chloroform, toluene, and MEK.

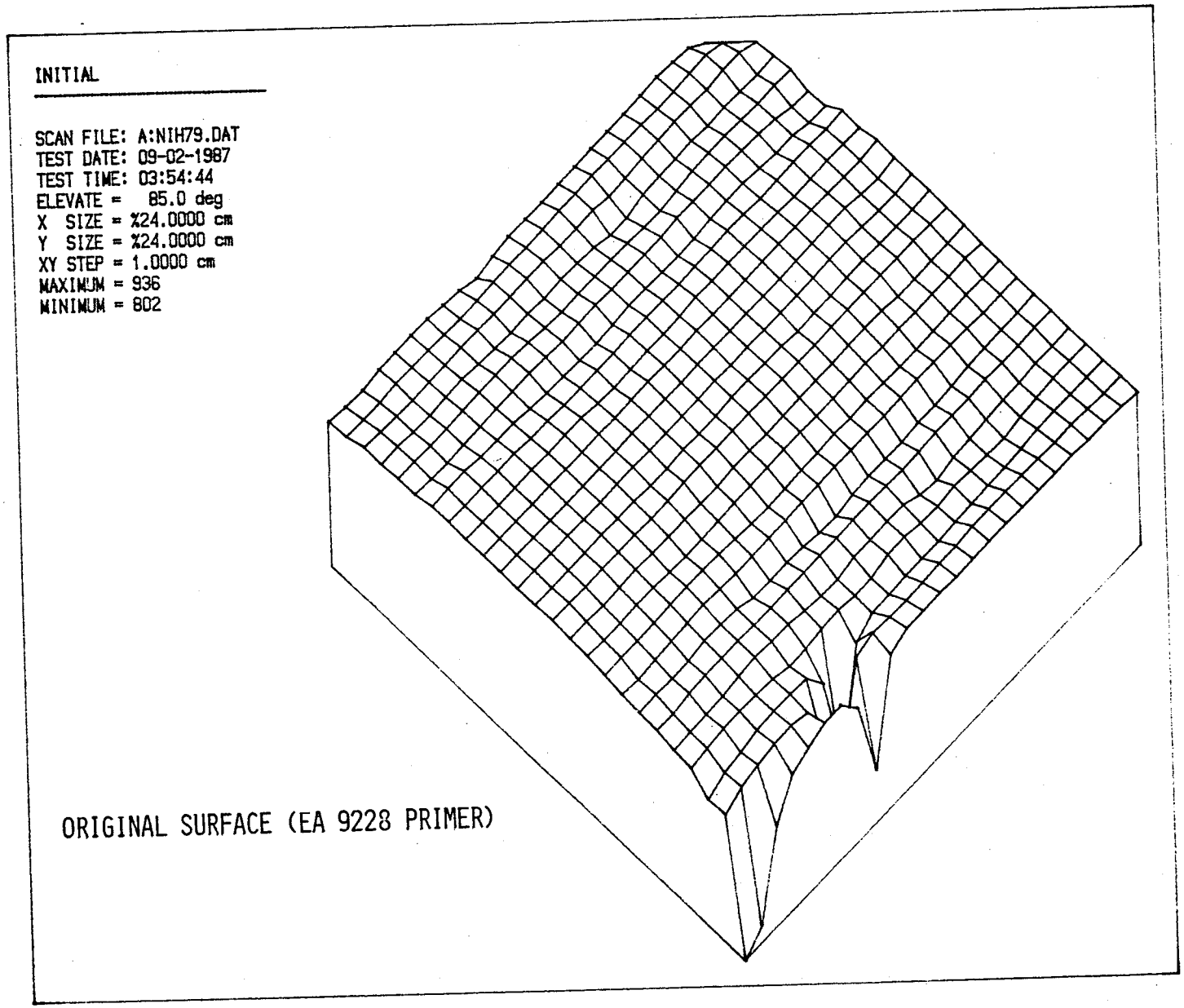

Figure 30. EA 9228 primer surface.

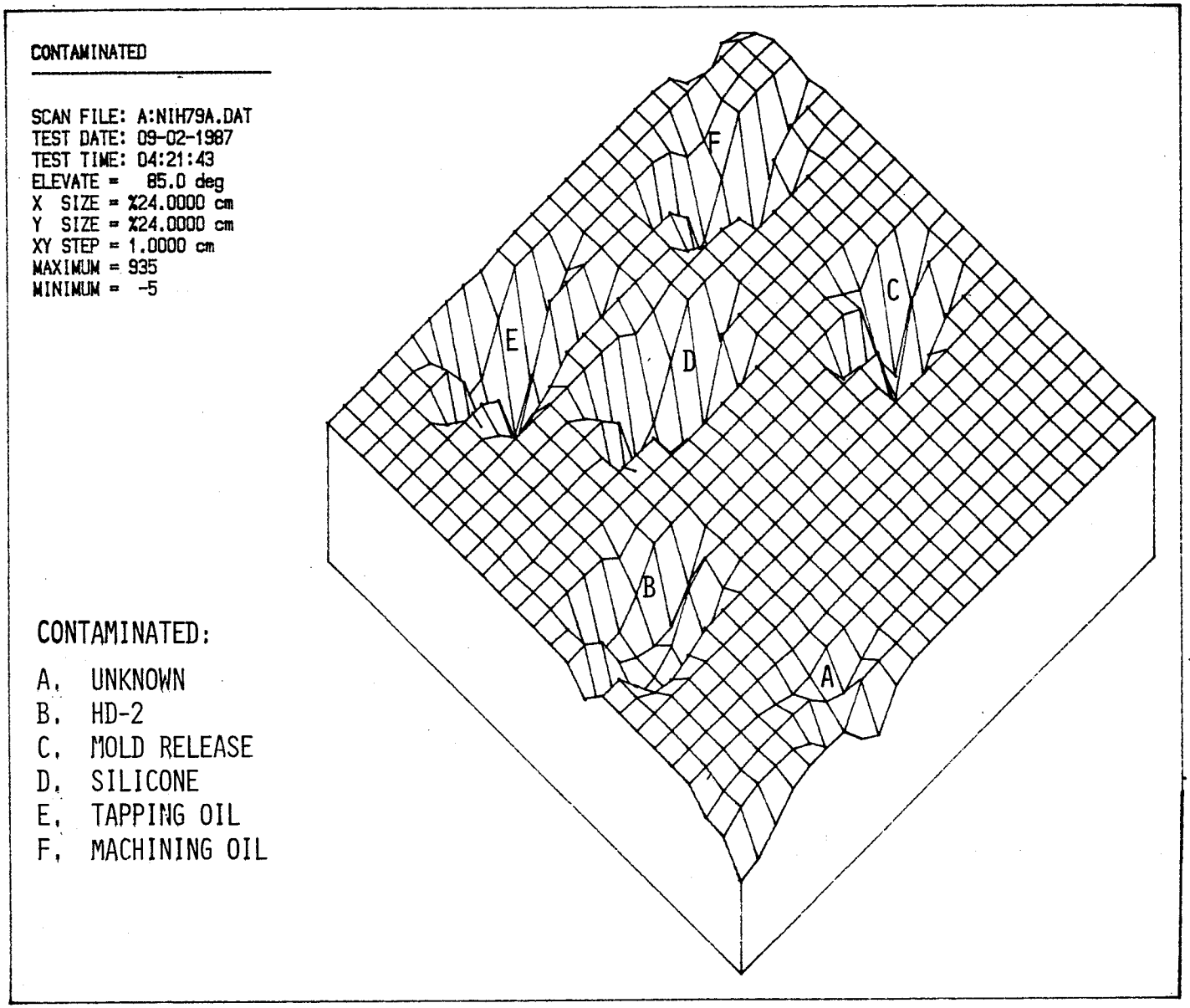

Figure 31. Contaminated primer surface.

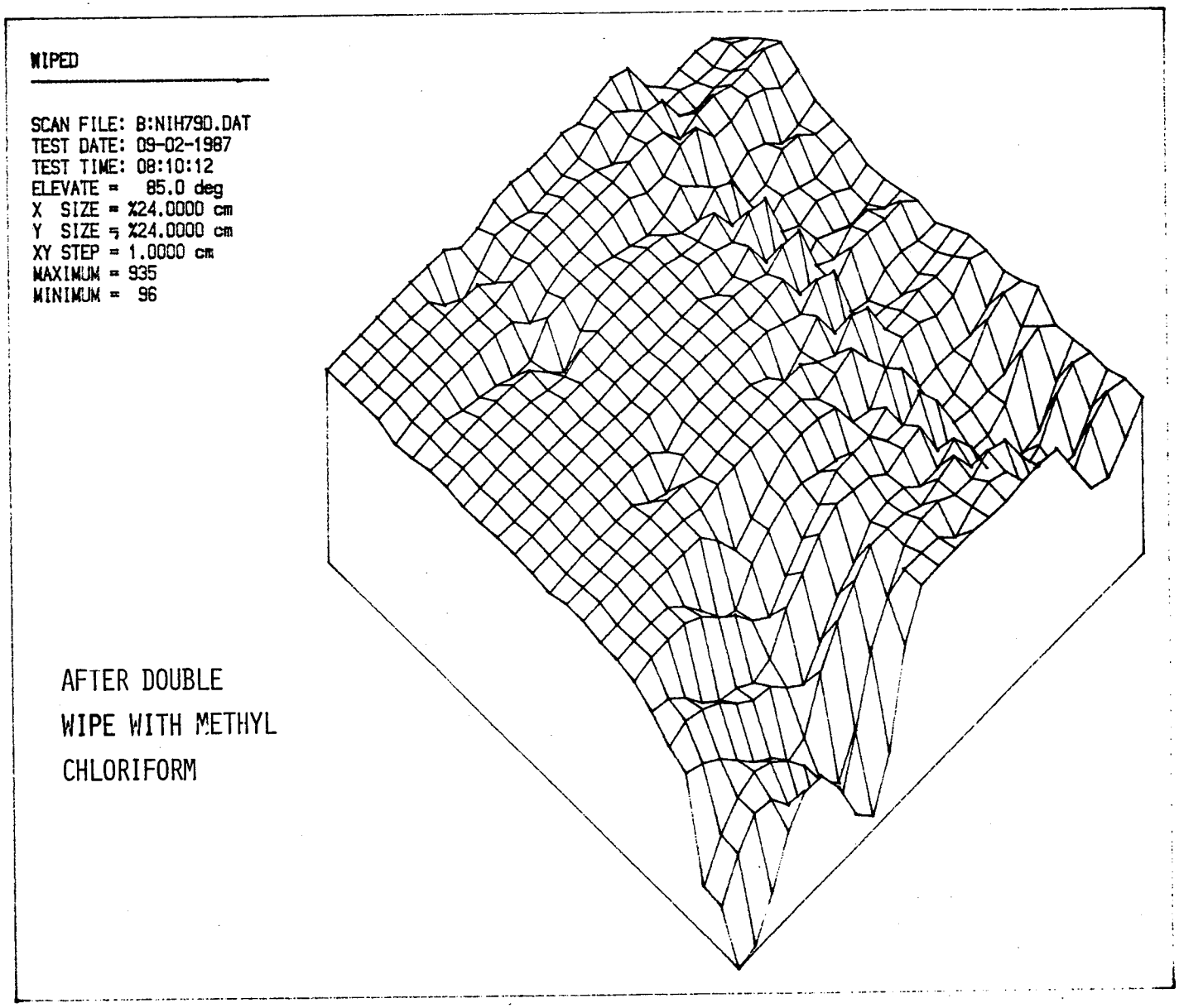

Figure 32. Effect of wiping contaminated primer surface.

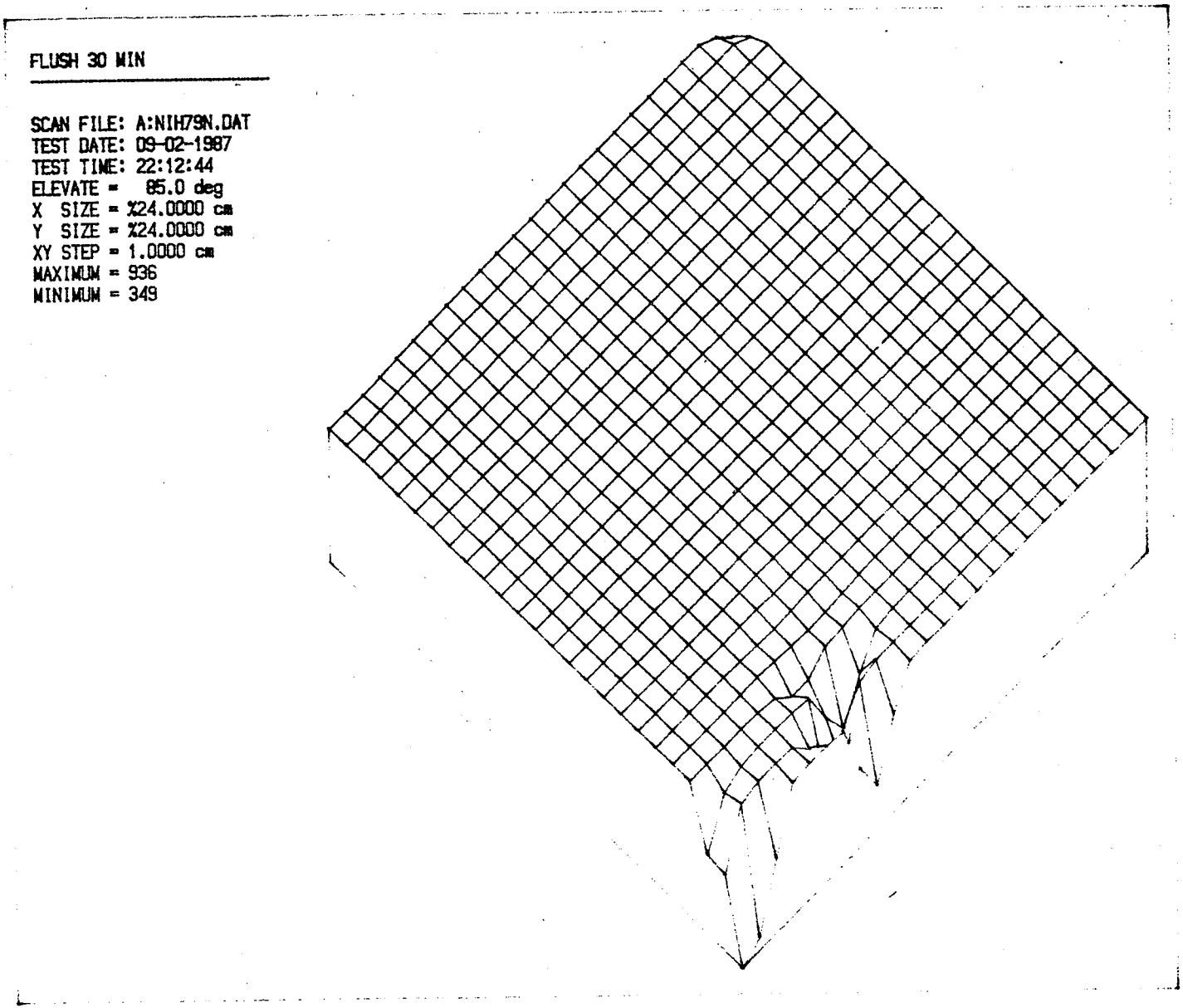

Figure 33. Effect of flushing contaminated primer surface.

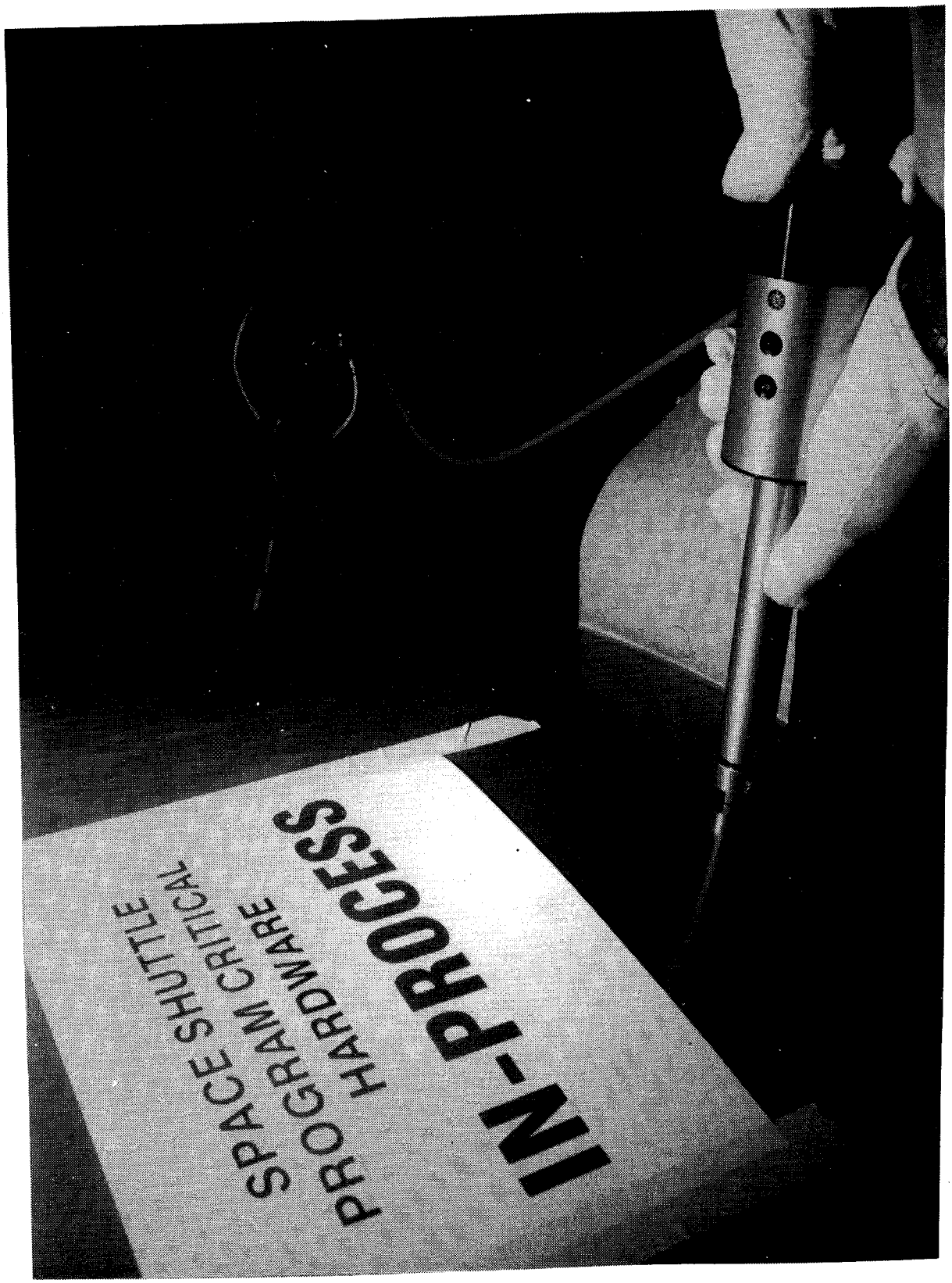

Figure 34. OSEE spot inspection of SRM nose inlet housing.

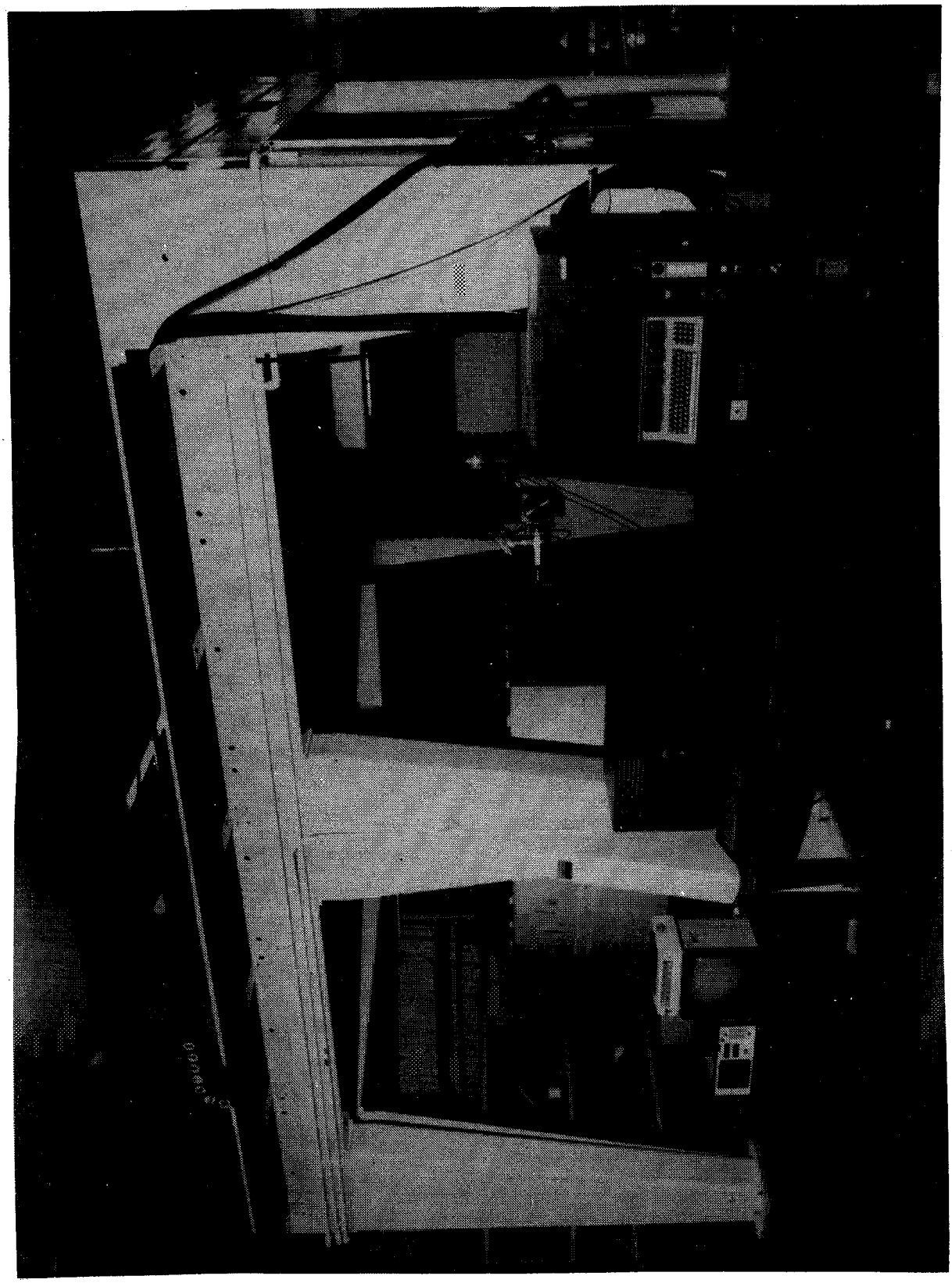

Figure 35. Gantry robot for scanning SRM nozzle parts using OSEE sensor.

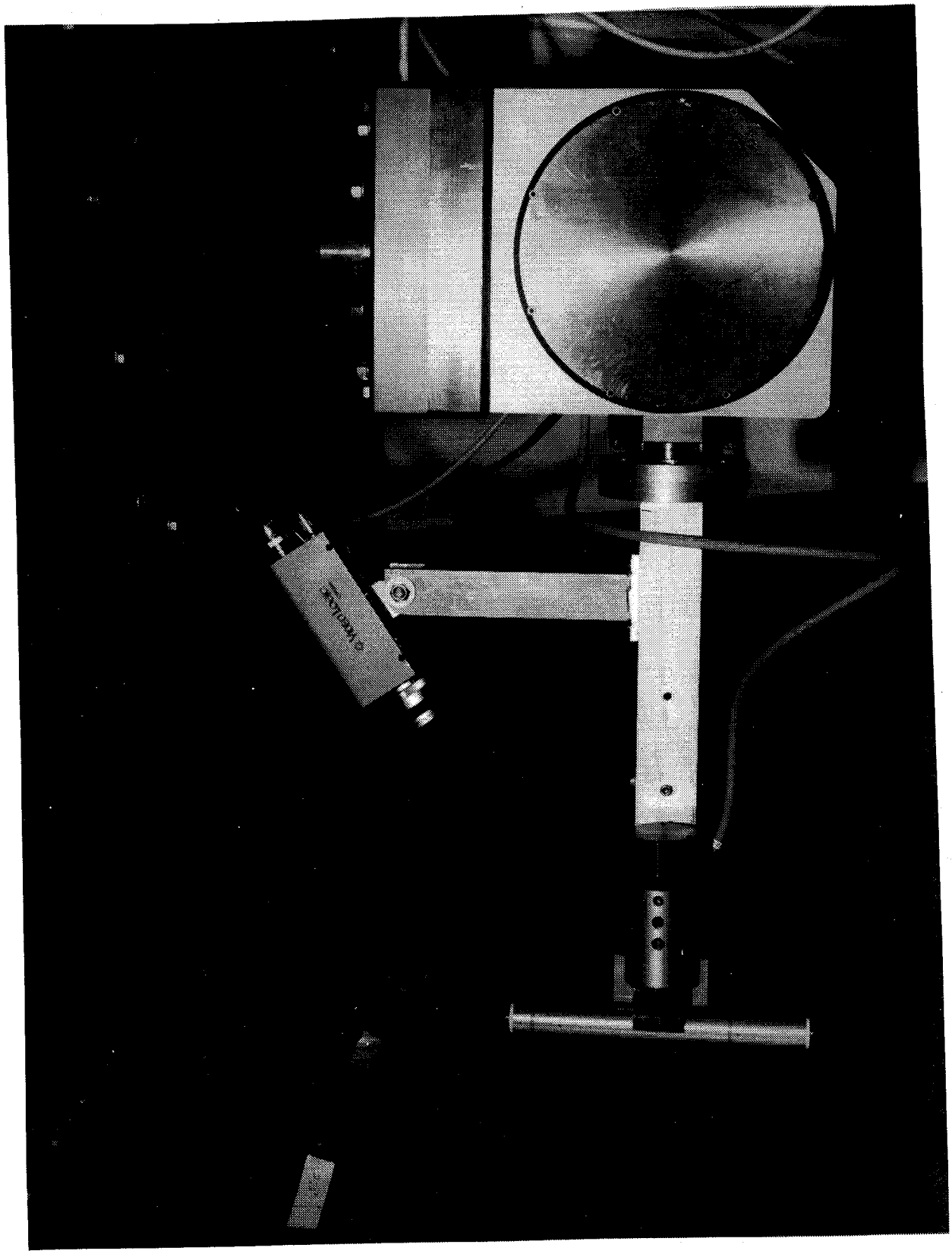

Figure 36. Robot arm with OSEE sensor.

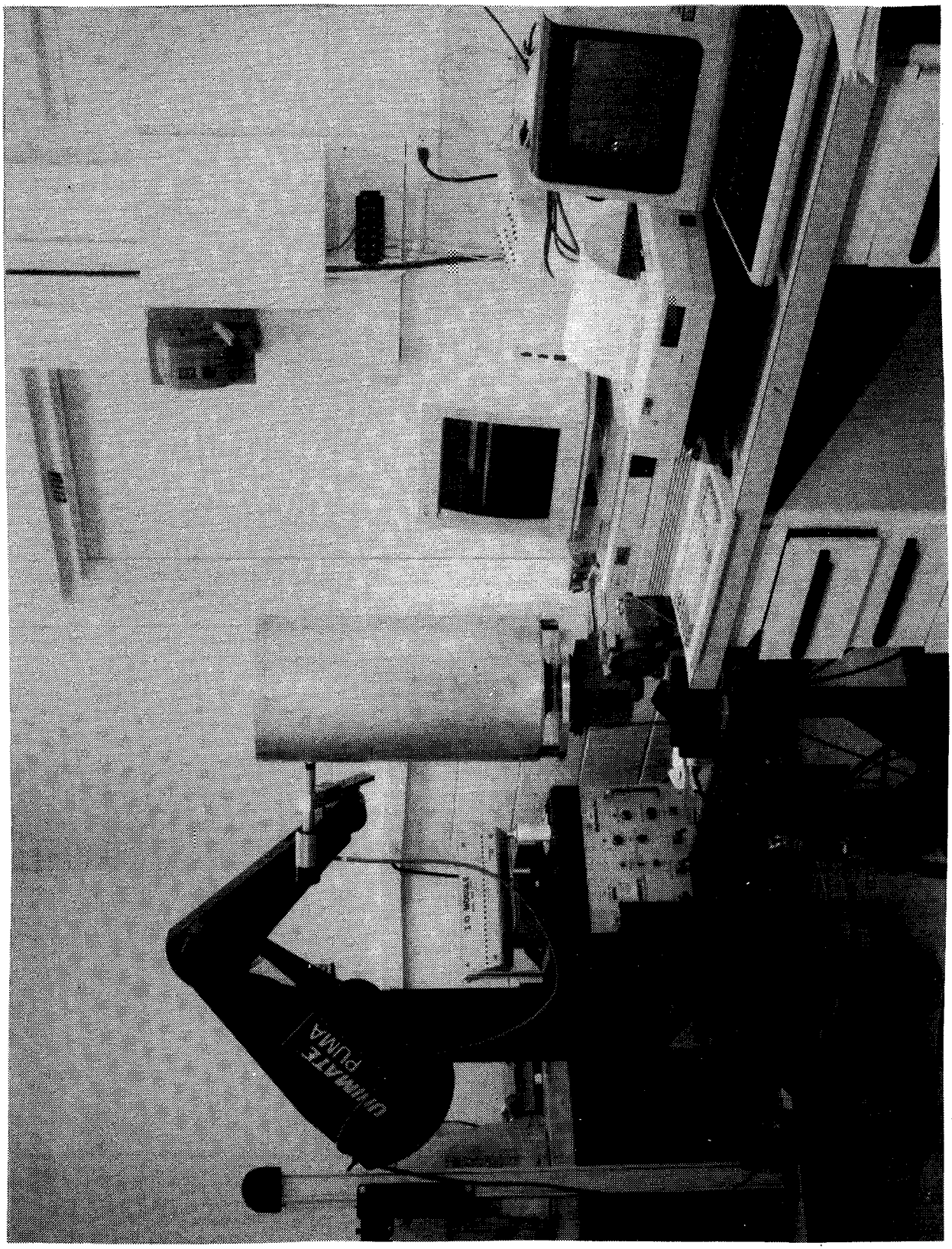

Figure 37. OSEE scanning of BSM.

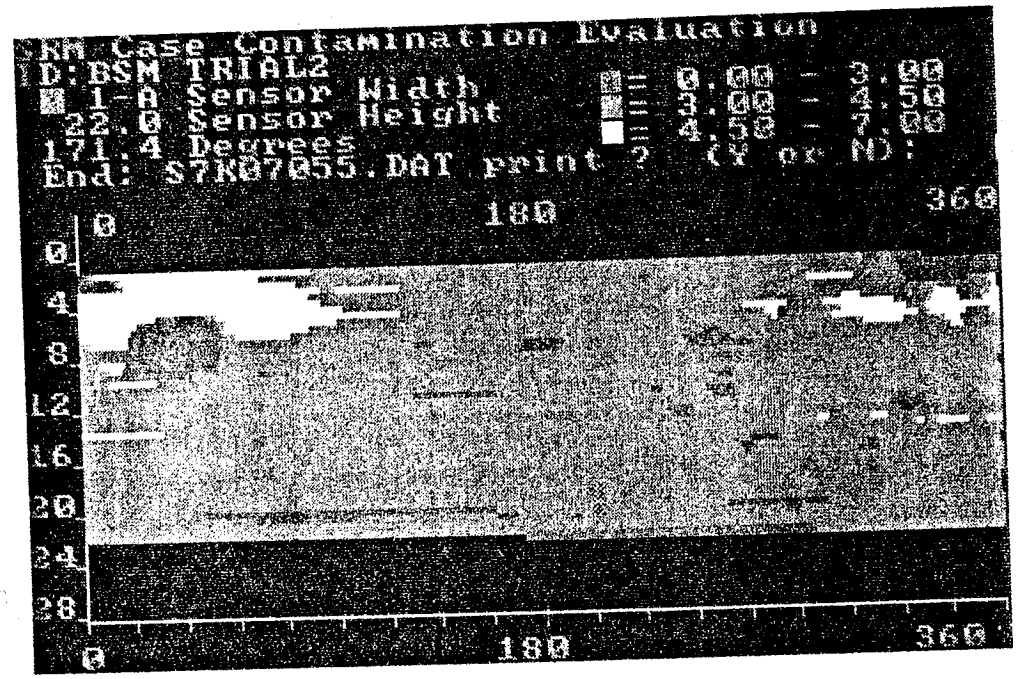

Figure 38. Scan of BSM Case before cleaning.

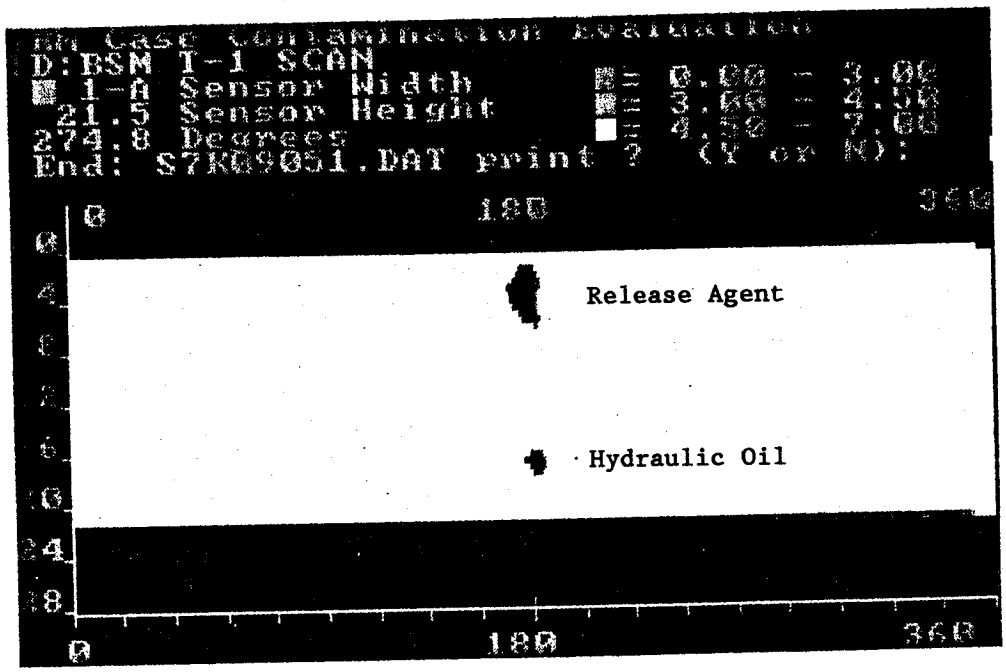

Figure 39. Scan of BSM Case after cleaning then contaminated.

The dark line at the bottom is due to the low signal obtained as the sensor moved away from the case before the scan was completed.

External Tank Studies

A task to establish quantitative OSEE data for External Tank bonding surfaces has been initiated. Currently the method for verifying cleanliness of these surfaces is the water break free test. Correlative data for OSEE versus the water break method will be obtained for various contaminant levels to establish relative sensitivities. In addition, the technique will be investigated as a tool for monitoring aging of surfaces prior to bonding.

CONCLUSIONS

It is believed, based on the work described above, that the photoelectron emission technique, properly applied, has the sensitivity and operational simplicity required to provide fast and cost effective surface contamination evaluations. Its application to bonding surface inspection for verifying established cleanliness requirements should result in the significant improvement of overall bond quality for a variety of bonding surfaces. NASA/MSFC plans to continue the development of this technique for a wide range of uses, including not only SRM, BSM, and ET hardware bonding surface inspection, but also for cure monitoring, location of impact damaged areas in composites, and other applications where knowledge of changes in surface characteristics can provide important insight as to the integrity of the material for its intended application.

REFERENCES

1. Smith, T.: Residual Silicone Detection. Final Report for NASA Contract NAS8-33694, SC5252.7FRD, Rockwell International Science Center, Thousand Oaks, CA, July 1980.

2. Bowen, R. and Minchey, J.: Systematic Contamination Survey. Final Report for NASA Contract NAS8-34307, Scientific Services, Inc., Huntsville, AL, September 1981.

3. Smith, T.: Quantitative Techniques for Monitoring Surface Contamination. In Surface Contamination - Genesis, Detection, and Control, K. L. Mittal (ed.), Phenum Press, New York, 1979.

4. Smith, T.: Photoelectron Emission from Aluminum and Nickel Measured in Air. Journal of Applied Physics, Vol. 46, No. 4, 1975, p. 1533.

5. Arora, A.: Surface Contamination Measurement and Control by Nondestructive Techniques. Journal of Environmental Sciences, Vol. 30, November-December 1985.

APPROVAL

A NONCONTACTING SCANNING PHOTOELECTRON EMISSION TECHNIQUE FOR BONDING SURFACE CLEANLINESS INSPECTION

By Dr. Raymond L. Gause

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

R. J. SCHWINGHAMER
Director, Materials and Processes Laboratory

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